AD/A-004 462

STORABILITY INVESTIGATIONS OF WATER LONG-TERM STORAGE EVALUATION

E. M. Vander Wall, et al

Aerojet Liquid Rocket Company

Prepared for:

Air Force Rocket Propulsion Laboratory

December 1974

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER: AFRPL-TR-74-76	2. GOVT ACCESSION NO.
3. RECIPIENT'S CATALOG NUMBER: AD/A-004462	
4. TITLE (and Subtitle): Storability Investigations of Water, Long-Term Storage Evaluation	5. TYPE OF REPORT & PERIOD COVERED: Annual
	6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s): Vander Wall, E. M., and Janser, G. R.	8. CONTRACT OR GRANT NUMBER(s): F04611-72-C-0062
9. PERFORMING ORGANIZATION NAME AND ADDRESS: Aerojet Liquid Rocket Company, Sacramento, California	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: 62302F, 3059, 11
11. CONTROLLING OFFICE NAME AND ADDRESS: Air Force Rocket Propulsion Laboratory, Edwards, California 93523	12. REPORT DATE: December 1974
	13. NUMBER OF PAGES: 65
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)	15. SECURITY CLASS (of this report): Unclassified
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for Public Release; Distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

High Purity Water, Stainless Steels, Inconel, Titanium, Particulate-Formation in Water, Effect of Microorganisms on Water Storability, Fluid/Material Compatibility, Biological Growth, Long-Term Storage of Water

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The objective of this program is to gather data that will permit the Air Force to assess the long term storage characteristics of water particularly with regard to formation of particulate matter, so that the feasibility of long-term storage of water for use as a transpiration coolant can be determined. Five metallic materials of construction are included in the program: 304 stainless steel, A-286 (aged) steel, 17-4 (aged) stainless steel, Inconel 718 (aged), 6Al-4V titanium (STA). Two types of water are used in

Abstract (cont.)

the program: oxygen-saturated, deionized, filtered, and oxygen-free, deionized, filtered. Five-year storage tests have been initiated in 304 and 17-4 PH stainless steels, A-286 steel, Inconel 718, and 6Al-4V titanium (STA) containers using the filtered, deionized waters.

Evaluation of water and containers stored for six-months and twelve-months has been completed. The data show that both oxygen-saturated and oxygen-free water can be stored in appropriate metal containers for the selected time periods without detrimental particulate matter formation or significant changes in the quality of the water. It is in excellent condition for transpiration coolant purposes.

FOREWORD

This report covers the work performed under Contract F04611-72-C-0062, "Storability Investigations of Water," performed by the Aerojet Liquid Rocket Company at Sacramento, California, and conducted under Air Force Project Task 305911 VD. The performance period covered from 15 August 1973 to 30 September 1974.

The project manager is Dr. S. D. Rosenberg; the project chemist is Dr. E. M. Vander Wall. The experimental work was conducted by Dr. Vander Wall; R. L. Beegle, Jr., senior chemist; J. A. Cabeal, senior laboratory technician; and G. R. Jansen, metallurgy specialist.

The program was administered under the direction of the Air Force Rocket Propulsion Laboratory, Mr. Oree Dyes, Project Engineer.

This report has been reviewed by the Information Office/DOZ and is releasable to the National Technical Information Service (NTIS). At NTIS it will be available to the general public including foreign nations.

Oree Dyes, GS-13
Project Engineer

Clark W. Hawk, GS-14
Chief, Engine Components Branch

FOR THE COMMANDER

Charles E. Sieber, Colonel, USAF
Chief, Liquid Rocket Division

TABLE OF CONTENTS

Preceding page blank

TABLE LIST

FIGURE LIST

FIGURE LIST (cont.)

SECTION I

INTRODUCTION

Inherent in the concept of transpiration-cooling is the requirement that the coolant remain free of particulate matter which may clog the passages of the cooling surface. The object of the "Storability Investigations of Water" program is to gather data that will permit the Air Force to assess the long term storage characteristics of water particularly with regard to formation of particulate matter. This report is the first annual progress report to document the experimental results from the six-month and twelve-month storage tests of water in selected metal containers for the five-year program being conducted under Contract F04611-72-C-0062.

The five metallic materials used for container material in the program are:

- 304 stainless steel
- A-286 (aged) steel
- 17-4 PH (aged) stainless steel
- Inconel 718 (aged)
- 6Al-4V Titanium (STA)

Two types of water are considered in the program:

- oxygen-saturated, deionized, filtered water and
- oxygen-free, deionized, filtered water

Three categories of tests are used for obtaining the data necessary for assessment of the storability of water. They are:

- Water Characterization
- Biological Characterization
- Container Examination

The investigations which led to the selection of the candidate metallic materials for tankage for use in the five-year storage of waters are reported in AFRPL-TR-73-94 "STORABILITY INVESTIGATIONS OF WATER, VOLUME I: EXPERIMENTAL STUDIES, FINAL REPORT, Aerojet Liquid Rocket Company, Sacramento, California, December 1973.

This annual report is presented in three sections: (1) Introduction, (2) Experimental Results and Discussions, and (3) Conclusions. In addition, there are two appendices provided for the convenience of the reader: Appendix A, Fabrication and Treatment Procedures for Water Containers which documents the history of the tanks; and Appendix B, Silting Index Measurement which describes a clogging tendency test.

SECTION II

EXPERIMENTAL RESULTS AND DISCUSSIONS

BACKGROUND INFORMATION

The purpose of the long-term storage tests is to demonstrate that water can be stored without formation of significant quantities of particulate matter and with insignificant corrosion of appropriate metal containers for time periods of at least five years in a controlled environment. The background information is presented for the convenience of the reader and documents the initial conditions of the selected containers and waters prior to the storage periods. The discussion is presented under the following headings: (1) Materials Selection, (2) Container Sterilization and Filling and (3) Storage Conditions.

A. MATERIALS SELECTION

1. Waters

Based on the data derived from the preceding experimental work (Reference 1) it was apparent that both oxygen-saturated and oxygen-free water were acceptable candidates for long-term storage tests. Further, the filtration of the water through a 0.22 micron pore size absolute filter was demonstrated to remove microorganisms effectively. Thus, the water used to fill the containers was passed through an activated charcoal bed to remove organic compounds and through two mixed-bed ion exchangers to obtain water that had an electrical resistance value of 1 megohm/cm or greater. The water was transferred through 0.22 micron filters into a 5 gallon stainless steel supply tank. To ensure the saturation of the water with oxygen, filtered oxygen was purged through the water in the supply tank for a minimum of 15 minutes. To obtain oxygen-free water, the water in the supply tank was heated to the boiling point of water for one hour while being purged with filtered, nitrogen obtained from the boil-off of liquid nitrogen. The tank was then pressurized with the filtered nitrogen, allowed to cool to ambient temperatures, and then repressurized with the filtered nitrogen. The outlet of the supply tank was fitted with a Twin 90 Filter* unit to assure the sterile characteristics of the water used to fill the storage containers.

Ref. 1 E. M. Vander Wall, R. E. Anderson, G. R. Janser, Storability Investigations of Water, Volume I, Experimental Studies, AFRPL-TR-73-94, Contract F04611-72-C-0062 (December 1973).

*A 0.22 micron pore size, absolute filter pack available from Millipore Corporation, Bedford, MA.

2. Metals

The selection of the materials of construction for the long term storage test containers was based on the results of the laboratory investigations (Reference 1). The aluminum alloys were eliminated from consideration due to their introduction of insoluble corrosion products which are a source of particulate matter in the water. Because test results on the remainder of the seven candidate materials were not discriminatory, choice was made on the basis of selecting not more than one alloy from each class of material. The one class of material with more than one representative was the 18% chromium - 8% nickel austenitic stainless steels, i.e., 304L, 347 and Arde-form 301. Hence two of these materials were eliminated to provide the five materials required for container fabrication. The 304L stainless steel was selected due to its attractiveness as an expulsion bladder material. Hence, the selected materials are: 304L stainless steel, A-286, 17-4PH stainless steel, Inconel 718 and 6Al-4V titanium. During fabrication, some 304 stainless steel parts were incorporated into the 304L stainless steel containers and consequently the containers are identified as 304 stainless steel containers. The fabrication procedures, the heat treatment cycles, the cleaning procedures and the passivation procedures to which the containers were subjected are presented in Appendix A of this report.

B. CONTAINER STERILIZATION AND FILLING

Following the final rinsing with filtered, deionized water, and subsequent drying of the containers in a vacuum chamber, the containers were wrapped with reusable sterilization paper. The wrapped containers were then sterilized in an autoclave at 250°F with 15 psig steam for 30 minutes followed by a 30-minute drying period. The containers were then stored in the paper to maintain their sterile condition.

All the steps required to fill the containers with water were conducted in a sterile, laminar-flow bench. The tanks were removed from the wrapping paper in the laminar flow bench. The tanks were weighed empty; then weighed when filled completely with sterile water to determine the total volume of the tank. The water was drained out and the tank was rinsed once more with the sterile water. A sample of the rinse water was checked for pH, conductivity, and Silting Index (see Appendix B). If the values indicated that particulate matter and dissolved species were not present, the tank was considered ready for filling; if the values indicated that contaminants were present, the tank was rinsed until there was no evidence of contamination. A Silting Index value of 1 or less for the rinse water when using the filter with a cross-sectional area of 1.0 mm^2 was used as the criterion that no significant quantity of loose particulate matter remained in the containers.

Before the final filling with oxygen-saturated deionized water, the tank was purged with oxygen from a filtered supply. The tank was then filled with the water and a sample was withdrawn for pH, conductivity, and Silting Index measurements. The ullage was adjusted to the ten percent value by weighing the container and its contents; the ullage space was purged with the filtered oxygen; and the container was capped with a sterile, tapered plug made from the same material as the container. The plug was seated in the fill-tube by use of a hammer. The containers were filled with the oxygen-free, deionized water in an analogous manner except that filtered nitrogen was used instead of oxygen for purging and blanketing the container.

The final sealing of the containers was accomplished by GTA welding the fill-tube/plug interface. The welds were inspected visually for any apparent anomalies. None were found. Then the containers were labeled for the long-term storage tests and placed in plastic bags.

The sampling plan for the long-term storage tests is to remove one container of each material with the two types of water for inspection and evaluation every six months for a period of five years. The contents will be characterized with respect to pH, conductivity, particulate content, and biological activity; and the containers themselves will be subjected to metallurgical examination if the other test data indicate that this is required.

C. STORAGE CONDITIONS

The storage area for the water containers is an air-conditioned room which is monitored continuously to document that the temperature is maintained at 70 ± 10°F and that the relative humidity is maintained at 50 ± 25 percent. The containers are stored in a closed metal cabinet to protect them from an accumulation of dust and the containers themselves are covered with plastic bags to prevent direct contact with foreign metal surfaces. The containers are visually examined on a weekly basis.

WATER CHARACTERIZATION

After six months of storage at the conditions defined above, and again after twelve months of storage, ten containers were removed for evaluation. They consisted of two containers of each selected material, one containing oxygen-free water and the other containing oxygen-saturated water.

A. PROCEDURES

After the six-month storage period, the water containers were washed with deionized water and then placed in a sterile, laminar-flow bench for further handling to remove the stored water. The outlet of the container was rinsed repeatedly with filtered, deionized water to remove any contaminants and then briefly subjected to a torch flame to sterilize the exterior of the metal. After the twelve month storage period, the water containers were immersed in a 95% ethanol bath prior to placement in the sterile, laminar-flow bench; and then after removal from the bath and placement in the flow bench, the residual alcohol on the tank surface was removed by burning. The outlet of the container was repeatedly exposed to a torch flame to assure a sterile condition. All the containers were opened in an identical manner. A sterile tubing cutter was used to sever the fill tube. The water was expelled from the containers by inserting into the fill tube of the container a sterile stainless steel capillary tube through which filtered, gaseous nitrogen was passed while the container itself was inverted. The first several ml of water were used to flush the tube and were discarded. Subsequent samples of water were collected for measurement of pH, electrical conductivity, dissolved solids, particulate matter, flow behavior, and for characterization with regard to possible biological contamination.

The measurement of the pH was made using a standard pH meter with a calomel reference electrode and a glass indicator electrode. The electrical conductivity of the water was measured using a Balsbaugh Conductivity Meter, Model No. 900-.01T with a standard dip cell. The dissolved solids content of the water was determined by evaporating 200-300 ml samples of the water to dryness and weighing the residue. In addition, any particulate matter which collected on the 0.8μ filter of the flow behavior device was examined microscopically and sized. The flow behavior of the water was evaluated using a Silting Index Apparatus (see Appendix B for description) which permits filtration of the liquid through a known area (1.0 mm^2) at a constant pressure so that the flow decay due to the presence of particulate matter may be recorded as a function of time. The standard method of the test is described in ASTM F52-69. The data are reported as a silting index values; the greater the value, the greater the degree of contamination by small particulate matter.

B. DISCUSSION OF RESULTS

The data obtained from the tests are presented in Table I. The data obtained during loading of the containers with the water initially are included in the tabulation to facilitate comparison and identification of trends. The baseline data are labeled as initial and the data from the six-month and twelve-month storage tests are labeled as final.

The significant items to note from the data are that: (1) there are generally slight increases in the pH values of the waters during storage in the containers; (2) as expected there is a general decrease in the resistance value of the waters due to an increase in concentration of ionic

TABLE I

DATA INDICATIVE OF THE STORABILITY OF WATER IN SELECTED METAL CONTAINERS

Container Material and Number	Exposure Period Months	Type of Water	Water Characterization: pH Initial	pH Final	Resistance megohm/cm Initial	Resistance megohm/cm Final	Silting Index Initial	Silting Index Final	Total Solids Content, mg/l Initial	Total Solids Content, mg/l Final	Biological Activity Initial	Biological Activity Final	Particulate Matter Characteristics
304SS, No. 3	6	O_2-free	7.0	7.2	1.75	0.84	0.23	1.06	<1	6	-	X	3.2 x 1.6μ platelets
304SS, No. 11	6	O_2-sat.	7.0	7.2	1.75	0.88	0.90	1.20	<1	<1	-	X	8 x 8μ platelets
304SS, No. 5	12	O_2-free	7.0	7.1	1.80	0.83	0.13	0.68	<1	<1	-	-	1 x 4μ platelets
304SS, No. 4	12	O_2-sat.	7.0	7.3	1.75	0.97	0.47	2.35	<1	<1	-	X	Mat of 3μ particles
A-286, No. 6	6	O_2-free	7.2	7.2	1.75	1.18	0	0.57	<1	<1	-	-	8 x 8μ platelets
A-286, No. 13	6	O_2-sat.	7.0	7.2	1.80	0.97	0.54	1.11	<1	<1	-	X	3.6μ platelets
A-286, No. A-3	12	O_2-free	7.2	7.7	1.80	1.35	0	0.67	<1	<1	-	X	1 x 3μ platelets
A-286, No. 19	12	O_2-sat.	6.9	6.7	1.80	1.08	0.46	0.65	<1	<1	-	-	3 x 3μ platelets
6Al-4V Ti, No. 2	6	O_2-free	7.0	7.4	1.90	0.84	0	0.69	<1	3.7	-	-	7.2 x 7.2μ → 5.4 x 5.4μ platelets
6Al-4V Ti, No. 1	6	O_2-sat.	7.0	7.2	1.70	1.05	0.86	1.80	<1	1	-	-	3.6 x 3.6μ platelets
6Al-4V Ti, No. 5	12	O_2-free	7.0	7.6	1.90	0.98	0.51	0.73	<1	<1	-	-	1 to 3μ particles, few 50μ agglomerates
6Al-4V Ti, No. 3	12	O_2-sat.	7.0	7.5	1.65	0.82	0.10	1.17	<1	<1	-	-	1 to 2μ particles agglomerated as a mat
Inconel 718, No. 1	6	O_2-free	7.0	7.6	1.70	0.58	0	0.42	<1	<1	-	-	1 to 2μ particles agglomerated to 30μ
Inconel 718, No. 2	6	O_2-sat.	7.0	7.4	1.70	0.95	0.28	0.83	<1	4	-	-	1 to 2μ particles agglomerated to 30μ
Inconel 718, No. 6	12	O_2-free	7.0	7.8	1.75	0.88	0	0.14	<1	<1	-	-	Mat of small particles, 18μ agglomerates
Inconel 718, No. 4	12	O_2-sat.	7.0	7.3	1.80	0.98	0	0.59	<1	<1	-	X	1.3μ particles, 50 x 90μ agglomerate
17-4PHSS, No. P-11	6	O_2-free	7.0	7.4	1.80	0.82	0	0.66	<1	<1	-	-	1 to 4μ particles agglomerated to 200μ
17-4PHSS, No. P-1	6	O_2-sat.	7.0	7.1	1.75	0.98	0	0.42	<1	<1	-	-	1 to 4μ particles agglomerated to 300μ
17-4PHSS, No. P-12	12	O_2-free	7.0	7.9	2.00	1.02	0.27	0.49	<1	<1	-	X	Mat of indeterminate sized particles
17-4PHSS, No. P-2	12	O_2-sat.	5.7	7.6	1.80	0.70	0.03	0.62	<1	<1	-	X	3 to 14μ platelets

species but the values indicate concentration levels which are equivalent to less than one part per million of metallic ions; (3) the Silting Index values indicate the presence of a slight amount of particulate matter in the water but the concentration levels are insignificant with regard to the quantities of particulate matter that are required to cause clogging in flow passages; (4) the total solids content of the water in most instances was below a mg/l which corresponds to the level of detection in the procedure used and the concentration level of solids in the waters which contained measurable solids was so low that the quality of the water is not impaired; and (5) the resistance values, Silting Index values, and total solids values are not significantly different between the six- and twelve-month storage periods.

The particulate matter that was collected on the filters appeared to be that which had continued to adhere to the container walls during the cleaning, pickling, passivation, and flushing procedures prior to fillings. In summation, the waters were all suitable for use in transpiration-coolant devices.

BIOLOGICAL CHARACTERIZATION

A. PROCEDURES

200 ml samples of the water taken from the storage containers were filtered through pre-sterilized filter pads which were transferred directly to sterilized Petri dishes containing suitable nutrients for direct colony counting after a suitable culturing period. The procedures are described in Standard Methods for Analysis of Water and Waste Water, American Public Health Association, 13th Edition (1971) and Biological Analysis of Water and Waste Water, AM 302, Millipore Corporation, Bedford, Mass. (1973). In addition, any biological organisms present were washed from the filter surfaces with a sterile buffer solution and placed directly in sterile nutrient solutions for culturing so that adequate samples are available for identifying the genus and the specific species of micro-organisms that might be present in the stored water. Based on the lag-period prior to growth of micro-organisms which have been observed earlier in the program (Reference 1), the tubes containing the nutrient solutions were incubated for periods up to one month.

B. DISCUSSION OF RESULTS

The results obtained by culturing samples from the water containers are presented in Table I under the heading "Biological Activity". The lack of any indications of micro-organisms being present is denoted by a minus sign; if growth was indicated in either the culture tube or on the filter pad, but not on both an "X" is used; and if growth was found in both the culture tube and on the filter pad a plus sign is used as an indication of the positive result. In no instance was a positive result obtained.

After a month of incubation of the samples from the six-month storage tests there was no indication of micro-organism growth on the pre-sterilized filter pads. Slight growth was observed in the culture tubes containing washings from the 304 stainless steel containers and one of the A-286 containers. The number of micro-organisms present was extremely small as indicated by the negative results with the filter pads and the slight amount of growth in the culture tubes. There was no evidence that any biological growth occurred during the six-month storage period. The micro-organisms found were identified as an Aeromonas species.

After a month of incubation of the samples from the twelve-month storage period, one of the culture tubes containing the washings from an Inconel 718 container exhibited growth, but the filter pad was negative. The micro-organisms present were identified as most likely being Pseudomonas aeruginosa. The filter pads used for the culturing the contents of both of the 17-4 pH containers, one of the 304 stainless steel containers, and one of the A-286 containers exhibited growth, but the corresponding culture tubes were all negative. The micro-organisms were identified as Pseudomonas species. Again there was no evidence that biological growth occurred during the twelve-month storage period in any of the containers.

The species that have been identified as being present in this random manner are isolated readily from water and from around sinks in laboratories. In reviewing the sampling procedure for the biological characterization, it appears that the procedure with repeated insertion of the cannula to expel water from the containers may be vulnerable to contamination by micro-organisms. The procedure is being modified so that future sampling will not be as susceptible to contamination.

In summation, the biological testing has shown that there is no evidence for any biological growth occurring during the storage periods.

CONTAINER EXAMINATION

A. PROCEDURES

After removal of the water from the containers by draining, they were vacuum dried for a day to insure sectioning in a dry condition. The containers were then photographed to document their general appearance. Sectioning of the containers to expose the internal surfaces was done by sawing without coolant to prevent contamination. Subsequent handling of the container halves was carefully performed to avoid touching the interior surfaces. The internal surfaces were then photographed to document their general appearance.

Further general examination at magnifications from 5 to 10X were conducted on all interior surfaces to further define conditions found in the aforementioned visual examination and to reveal additional suspect areas. Selected discrepancies were then identified for additional examination at magnifications to 40X. All welds were examined at 40X magnification. Representative discrepancies were photographed at magnifications adequate for defect definition. Those defects requiring further definition were examined metallographically to establish their cause and extent. Sections taken either through or immediately adjacent to the affected area were mounted, polished, and examined. Photomicrographs were taken to document the condition. Contaminants or corrosion products capable of being sampled were analyzed by X-ray diffraction and emission spectrographic techniques to establish their composition. All interior surfaces were dye-penetrant inspected to determine whether any defects were undetected during the visual examinations. No additional indications of defects were found.

B. DISCUSSION OF RESULTS

The results of the container examinations are discussed under three headings: (1) General Visual Examination, (2) Metallographic and Chemical Analyses, and (3) Implications of the Results of the Examinations.

1. General Visual Examination

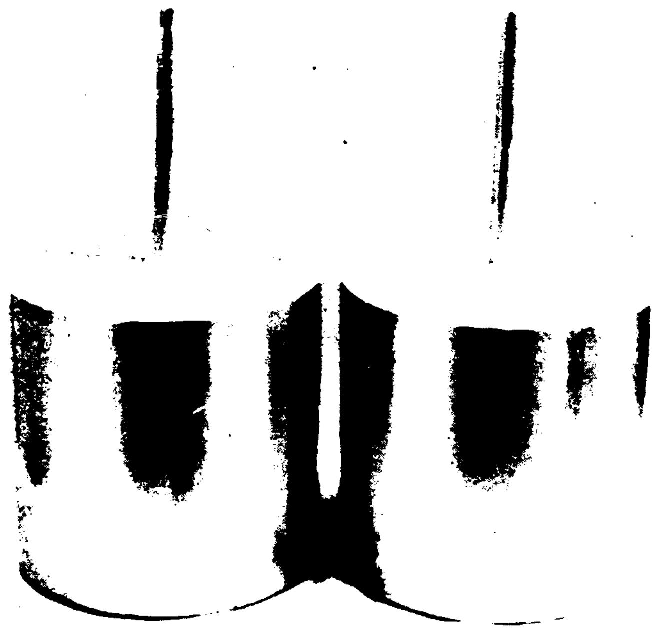
The external appearance of the containers is documented photographically in Figures 1 through 5. The internal appearance of the containers is documented photographically in Figures 6 through 15. Examination of these surfaces without visual aids showed full penetration for the full length of all weldments. Other conditions resulting from fabrication and cleaning procedures were: (1) etching of A-286 containers during pickling; (2) isolated dark areas, a tightly adherent smut, and isolated shiny deposits of material in the 17-4pH stainless steel containers; (3) a tightly adherent smut in the Inconel 718 containers; and (4) isolated areas of residual titanium oxide, localized circular areas of attack, and a general mottled appearance including fingerprint contamination in the 6Al-4V titanium containers.

No difference could be determined between the 6-month and 12-month exposure containers, or between those holding the oxygen-free and oxygen-saturated water.

2. Metallographic and Chemical Analyses

A summary of the analyses performed on the containers is presented in Table II. The results of macro- and microexamination are shown

6 MONTHS

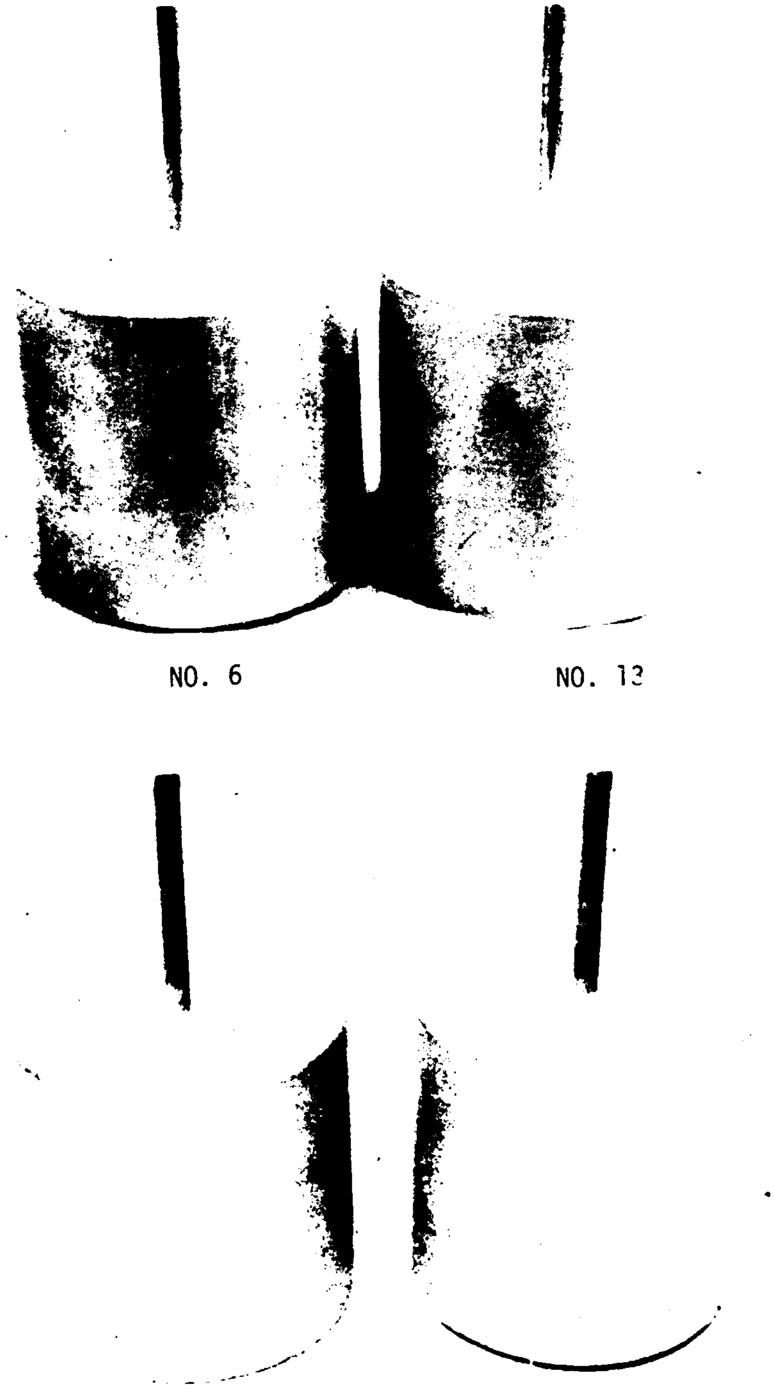


NO. 3 NO. 11

12 MONTHS

NO. 4 NO. 5

Figure 1. 304 Stainless Steel Containers, Magnification 1/3X



6 MONTHS

NO. 6

NO. 13

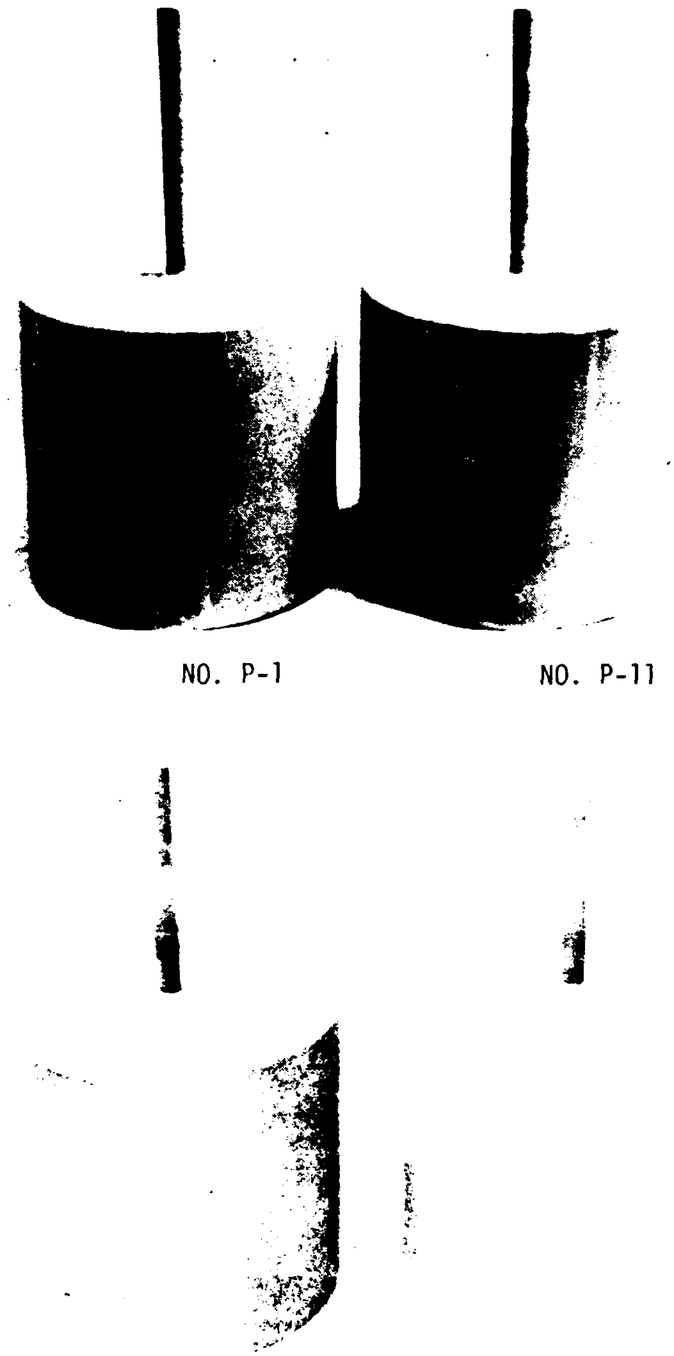
12 MONTHS

NO. 19

NO. 3

Figure, 2. A-286 Containers, Magnification 1/3X

6 MONTHS



NO. P-1 NO. P-11

12 MONTHS

NO. P-12 NO. P-2

Figure 3. 17-4PH H-1025 Stainless Steel Containers, Magnification 1/3X

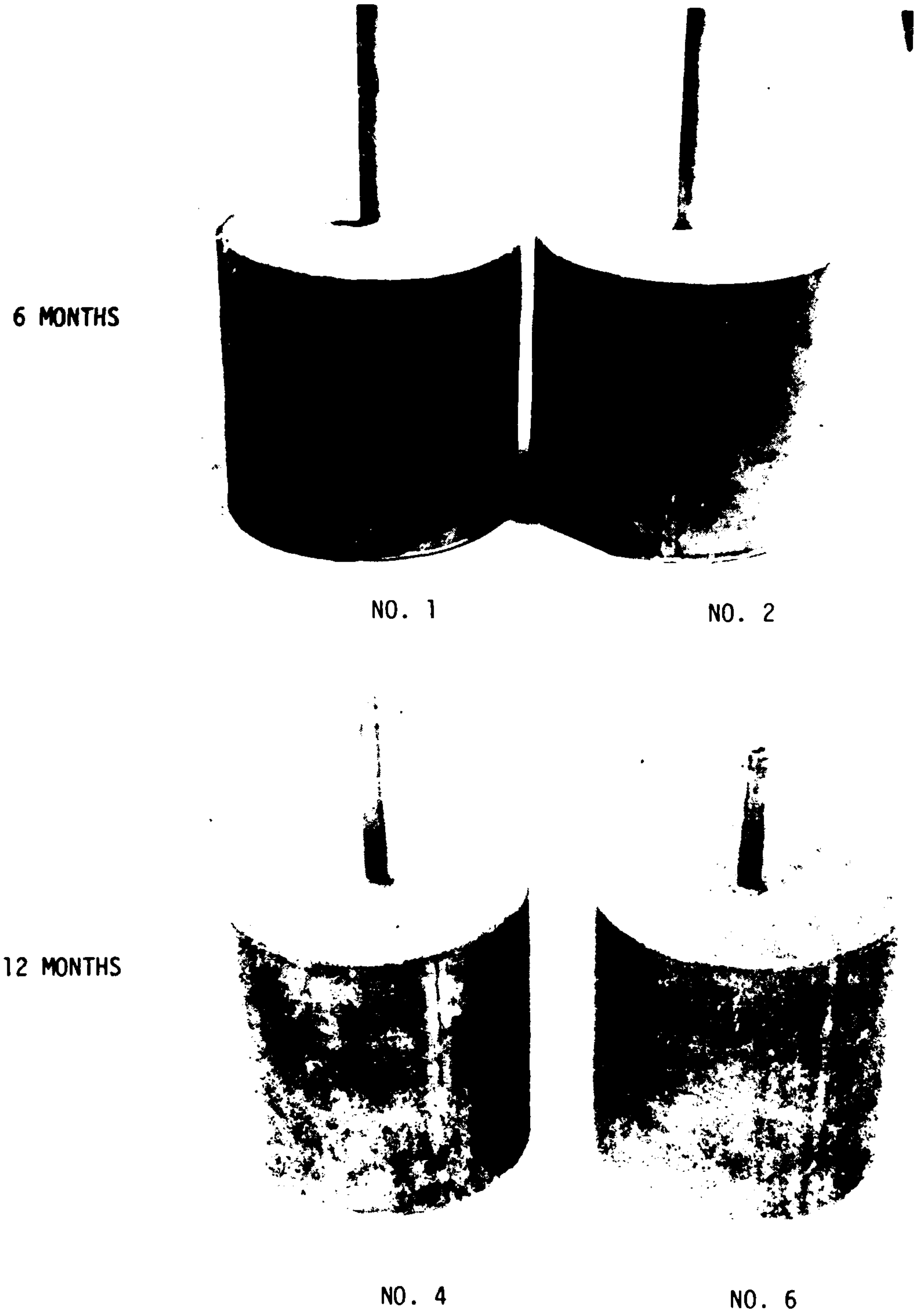
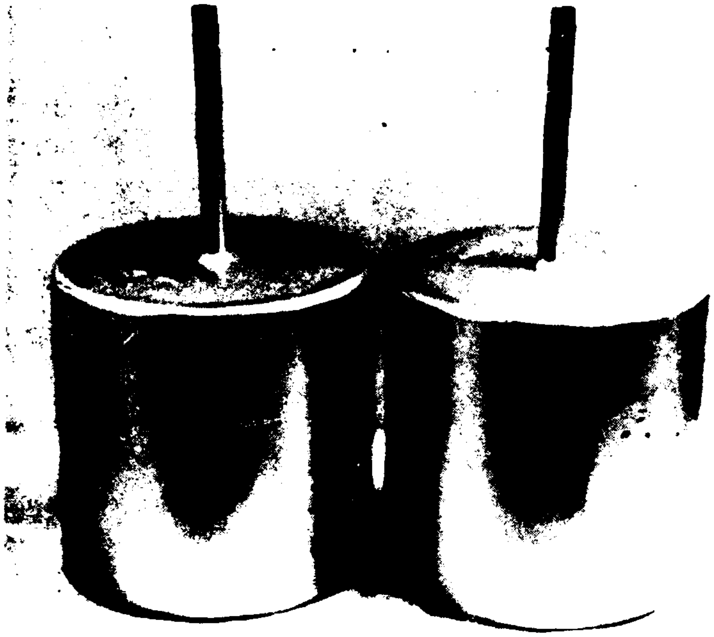


Figure 4. Inconel 718 Containers, Magnification 1/3X

6 MONTHS

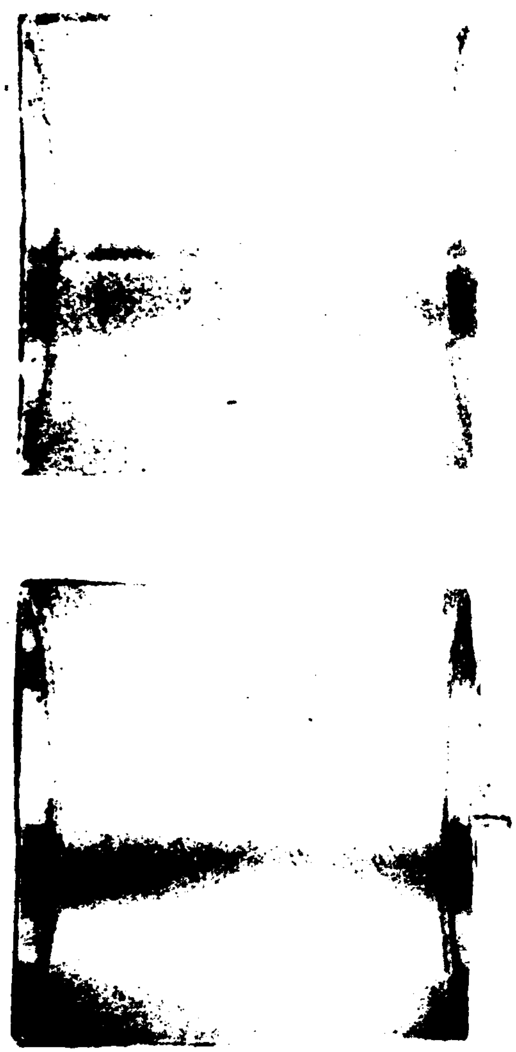


NO. 1 NO. 2

12 MONTHS

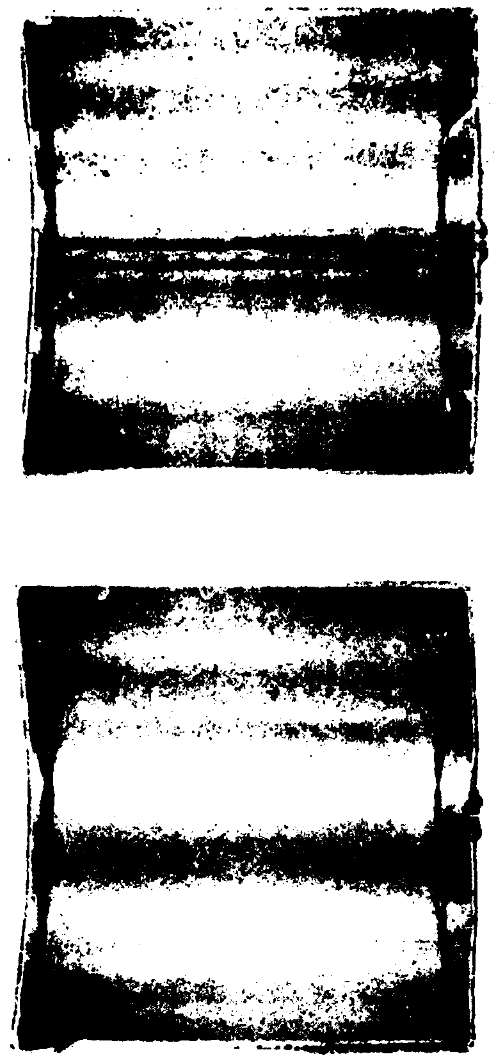
NO. 3 NO. 5

Figure 5. 6Al-4V Titanium Containers, Magnification 1/3X



NO. 3

6 MONTHS

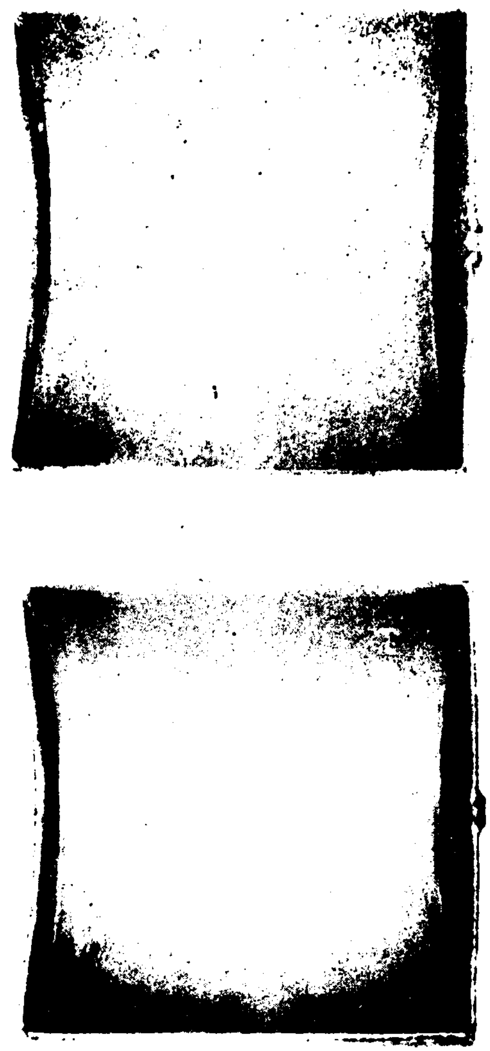


NO. 5

12 MONTHS

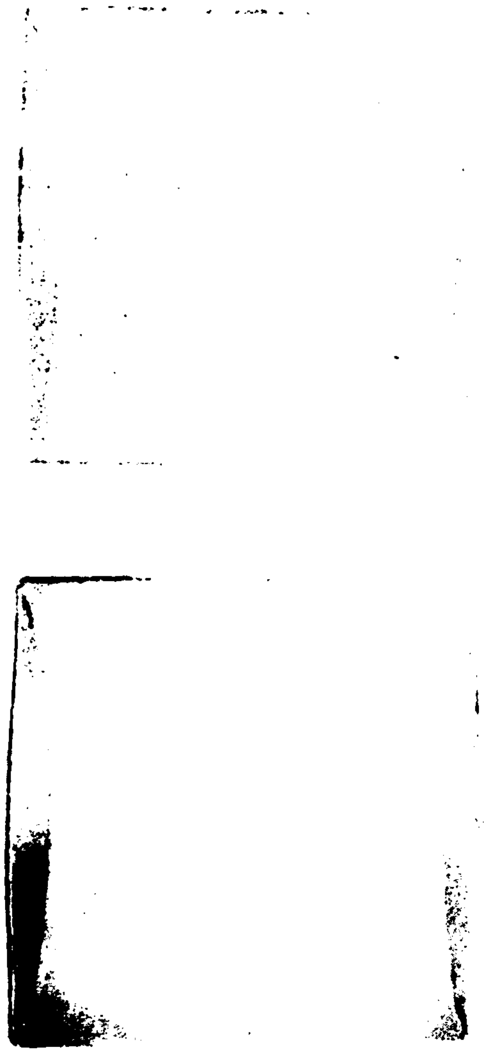
Figure 6. Interior of 304 Stainless Steel Containers after Exposure in Oxygen Free Water, Magnification 3/5X

NO. 11
6 MONTHS

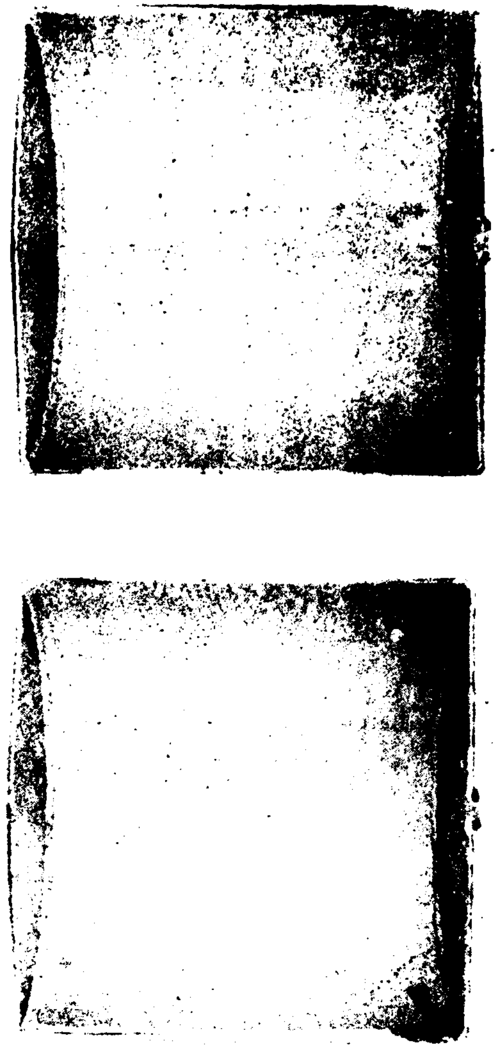


NO. 4
12 MONTHS

Figure 7. Interior of 304 Stainless Steel Containers After Exposure in Oxygen Saturated Water, Magnification 3/5X

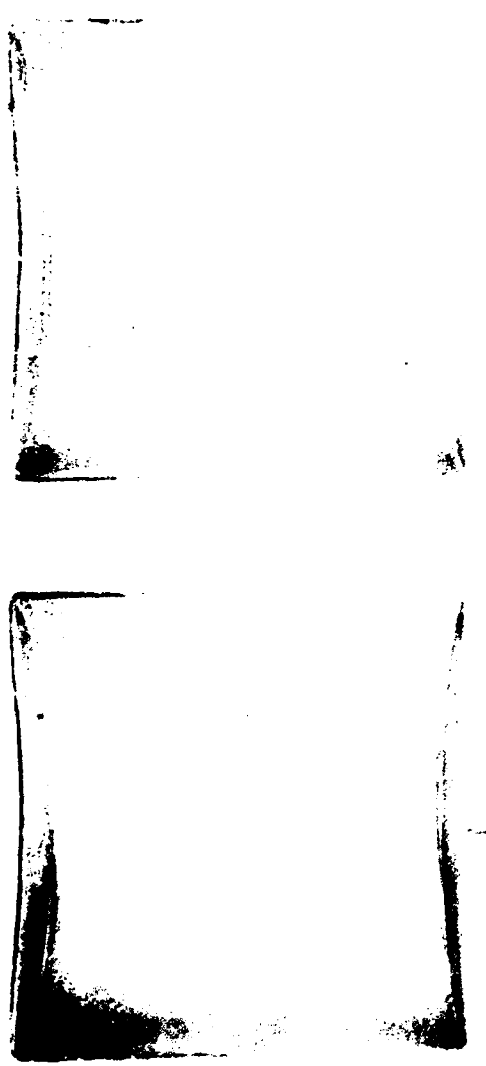


NO. 6
6 MONTHS

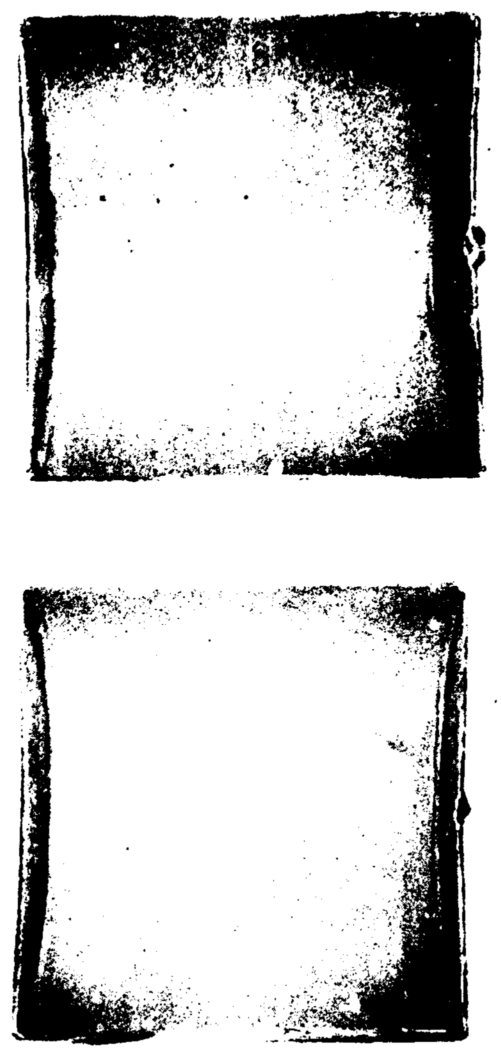


NO. A-3
12 MONTHS

Figure 8. Interior of A-286 Containers After Exposure in Oxygen Free Water, Magnification 3/5X

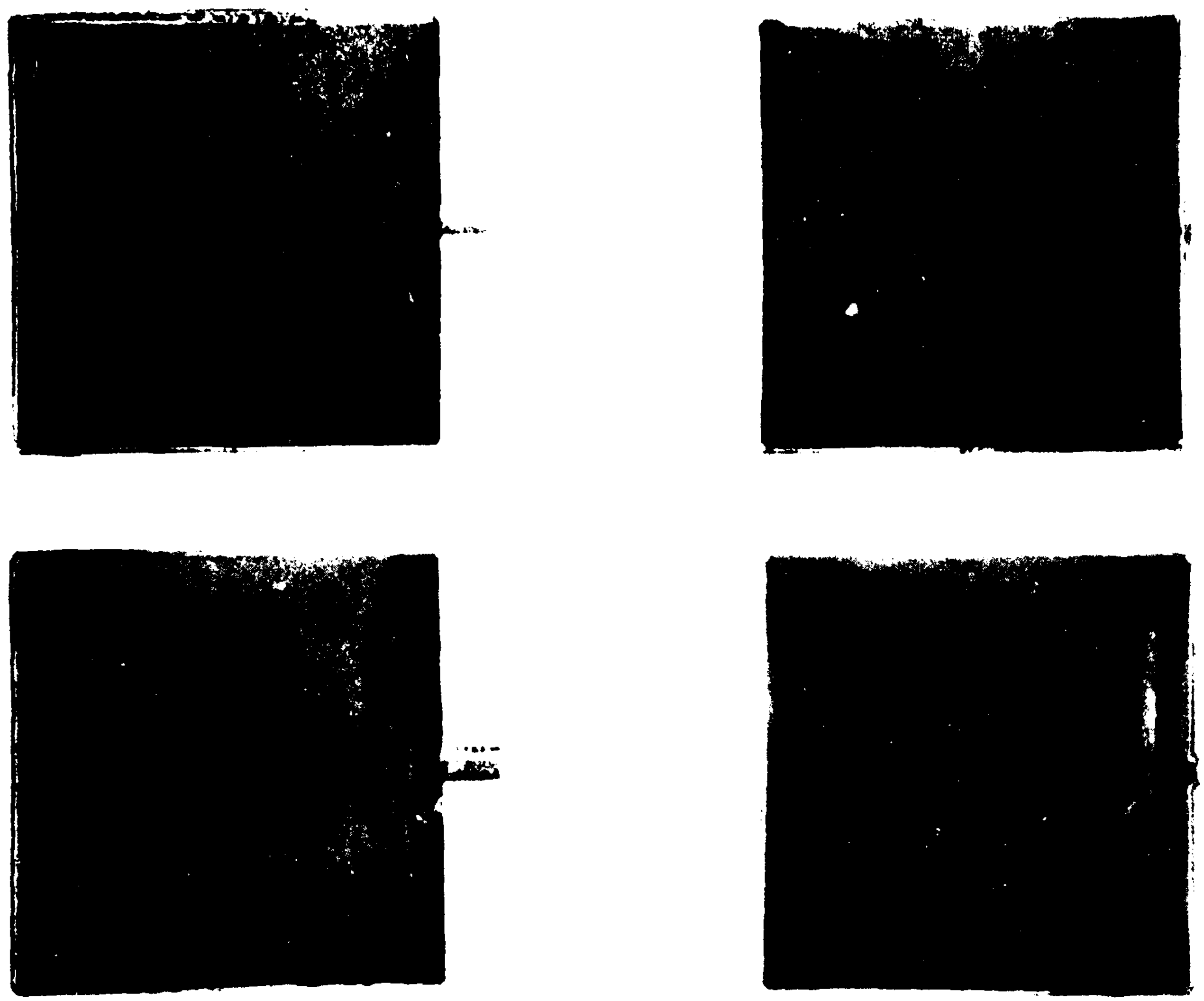


NO. 13
6 MONTHS



NO. 19
12 MONTHS

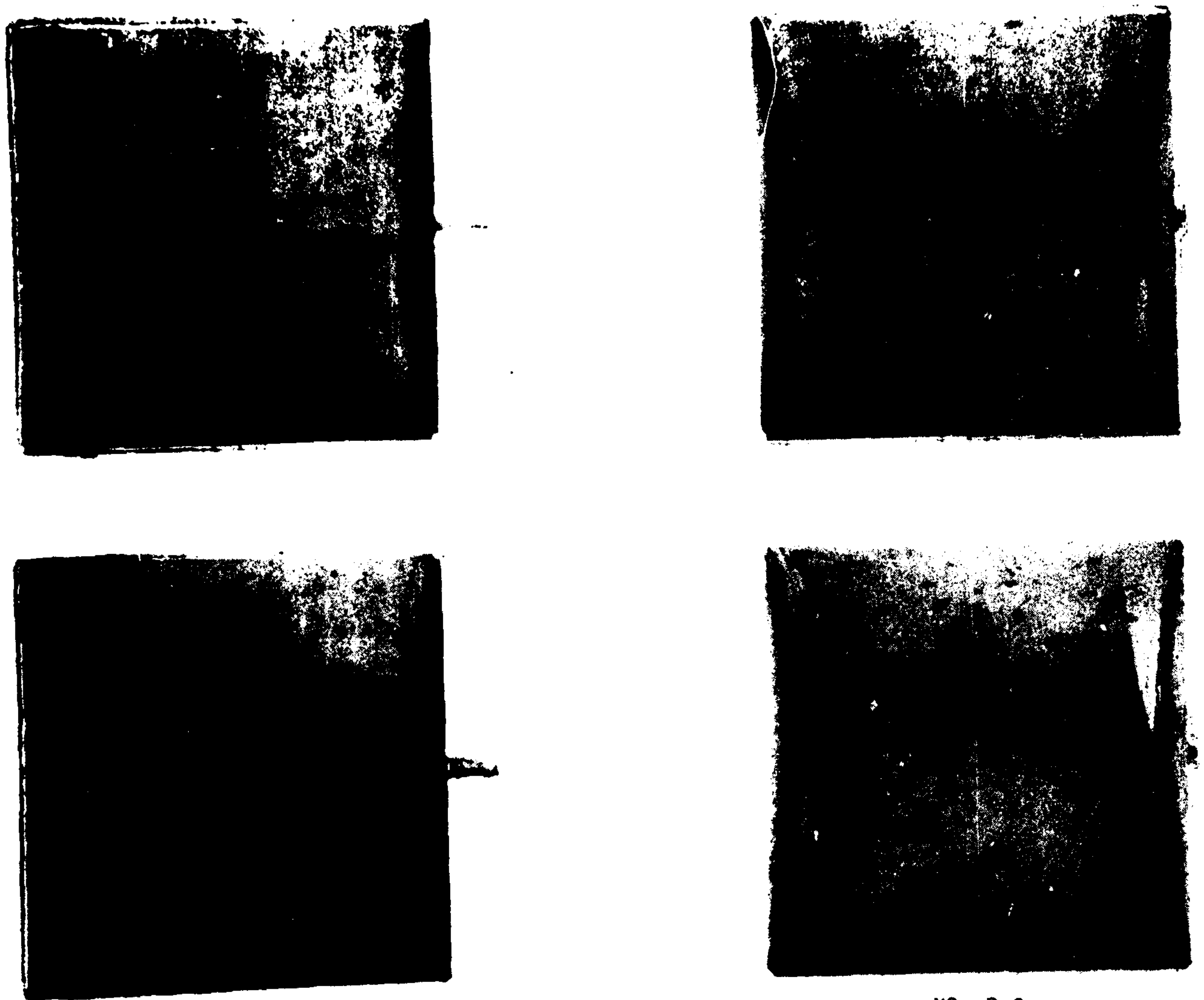
Figure 9. Interior of A-286 Containers After Exposure in Oxygen Saturated Water, Magnification 3/5X



NO. P-11
6 MONTHS

NO. P-12
12 MONTHS

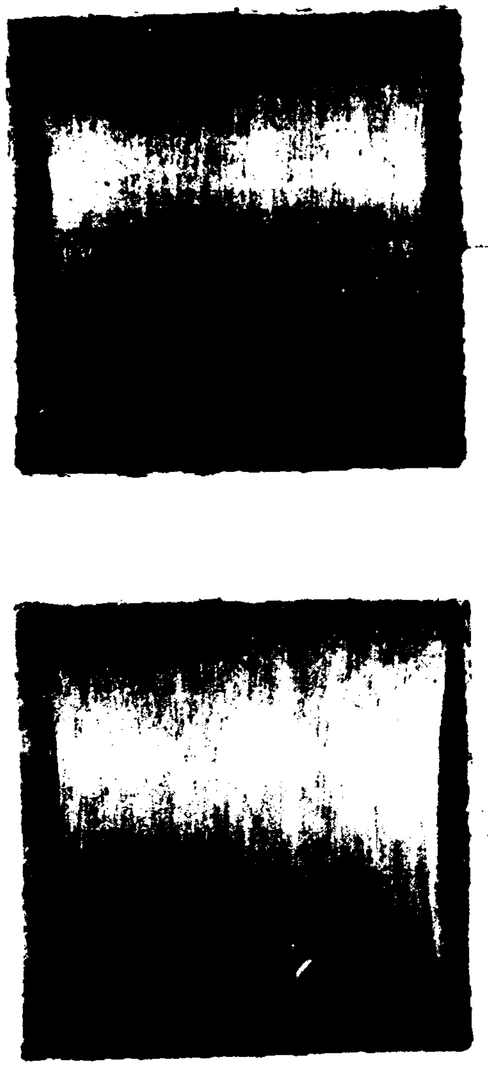
Figure 10. Interior of 17-4PH H-1025 Stainless Steel Containers After Storage in Oxygen Free Water, Magnification 3/5X



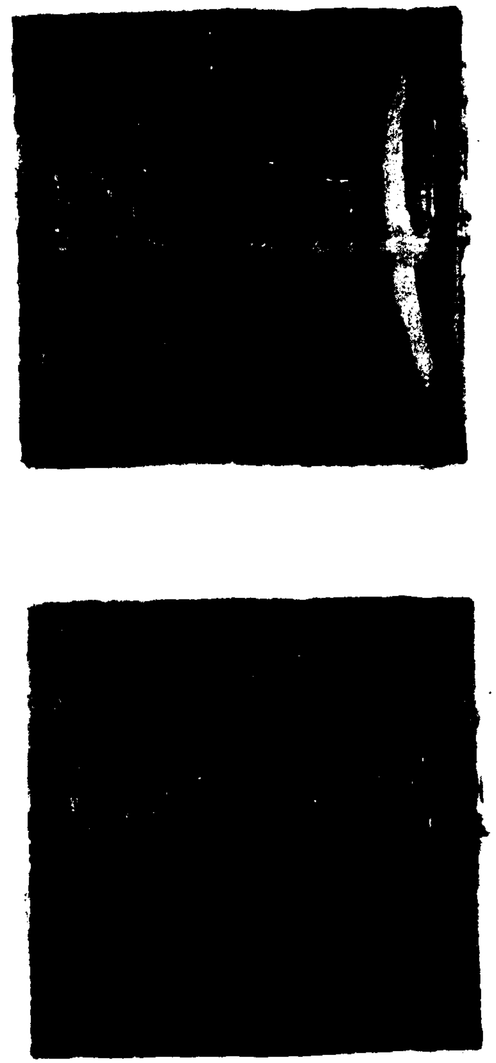
NO P-1
6 MONTHS

NO. P-2
12 MONTHS

Figure 11. Interior of 17-4PH H-1025 Stainless Steel Containers after Exposure in Oxygen Saturated Water, Magnification 3/5X

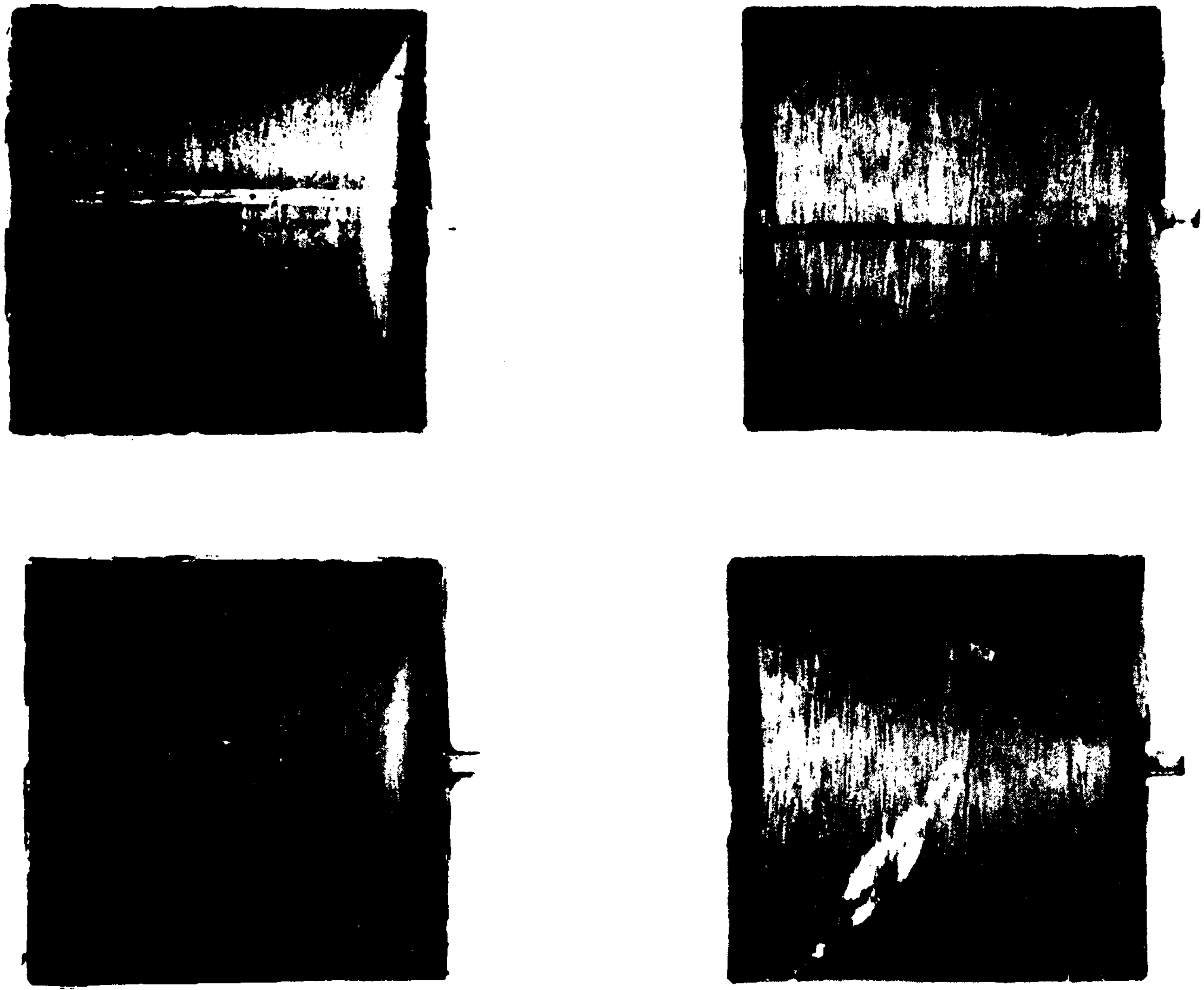


NO. 1
6 MONTHS



NO. 6
12 MONTHS

Figure 12. Interior of Inconel 718 Containers After Exposure in Oxygen Free Water, Magnification 3/5X



NO. 2
6 MONTHS

NO. 4
12 MONTHS

Figure 13. Interior of Inconel 718 Containers After Exposure in Oxygen Saturated Water, Magnification 3/5X

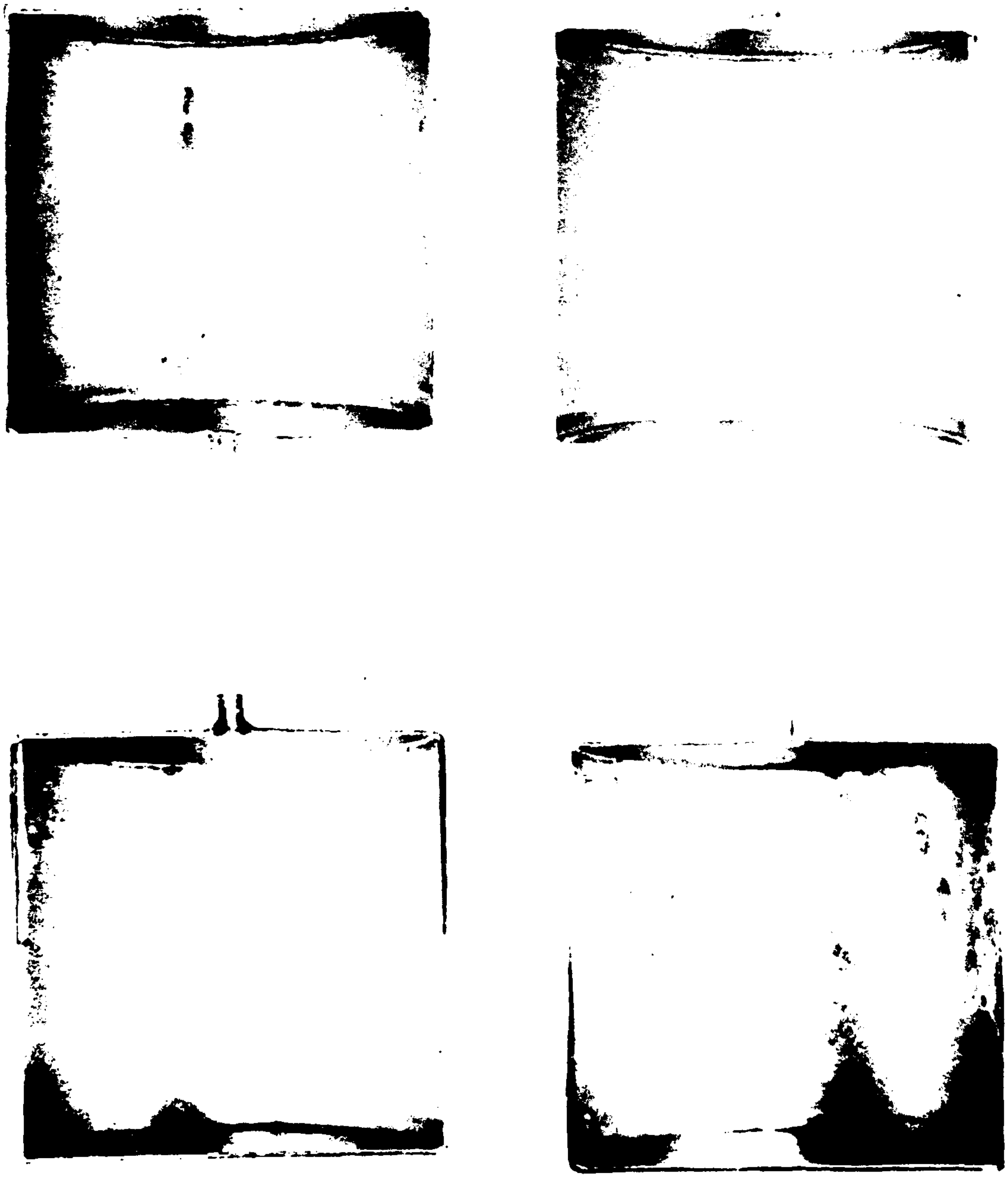
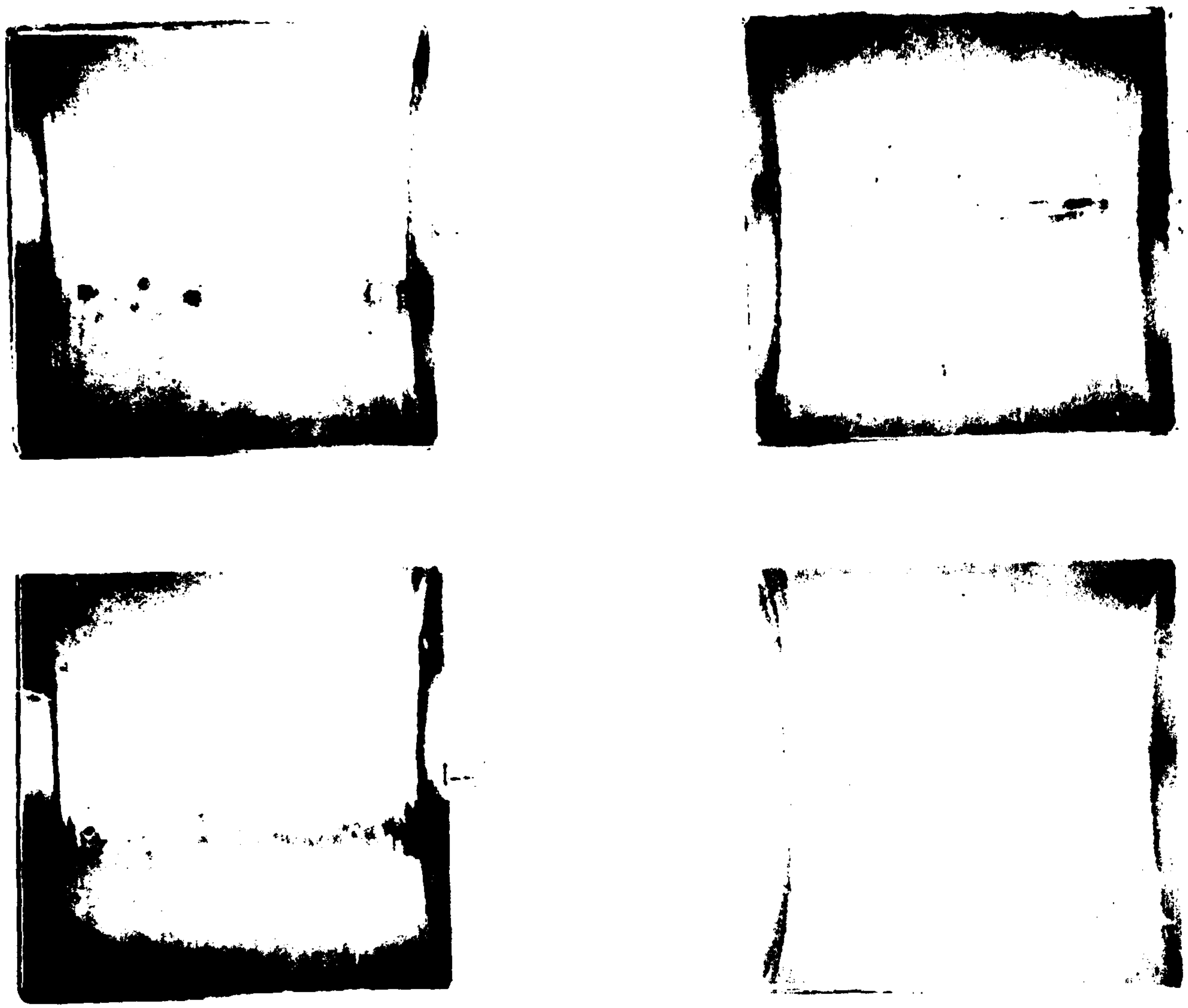


Figure 14. Interior of 6Al-4V Titanium Alloy Containers After Exposure to Oxygen Free Water, Magnification 3/5X



NO. 1
6 MONTHS

NO. 3
12 MONTHS

Figure 15. Interior of 6Al-4V Titanium Alloy Containers After Exposure in Oxygen Saturated Water, Magnification 3/5X

TABLE II

SUMMARY OF CONTAINER ANALYSES

Material	Tank Ident.	Exposure Period Months	5-10X Inspection	40X Inspection	40X Weld Inspection	Chemical Analysis	Dye Penetrant Inspection	Metallographic Documentation Macro	Metallographic Documentation Micro
304 SS	3	6	-	-	Light green refractory contaminant in weld	-	No indications*	X	X
304 SS	3W	6	-	-	-	-	No indications*	-	-
304 SS	11	6	Light red stain near closure weld	Light red stain near closure weld	-	-	No indications*	X	-
304 SS	11W	6	-	-	-	-	No indications*	-	-
304 SS	4	12	-	-	-	-	No indications*	-	-
304 SS	4W	12	-	-	-	-	No indications*	-	-
304 SS	5	12	-	-	-	-	No indications*	-	-
304 SS	5W	12	-	-	Light green refractory contaminant in weld	-	No indications*	X	X
A-286	13	6	Weld crack, end closure	-	Weld crack, end closure	-	No indications*	X	X
A-286	13W	6	-	-	-	-	No indications*	-	-
A-286	6	6	2 Weld cracks, end closure	-	2 Weld cracks, end closure	-	No indications*	X	X
A-286	6W	6	-	-	-	-	No indications*	-	-
A-286	A-3	12	Weld crack, end closure	-	Weld crack, end closure	-	No indications*	X	X
A-286	A-3W	12	-	-	-	-	No indications*	-	-
A-286	19	12	-	-	-	-	-	-	-
A-286	19W	12	-	-	-	-	-	-	-
17-4PH SS	P-11	6	Rough surface where heat treat scale removed by pickling	Metallic granular appearing contamination	Rough surface where heat treat scale removed by pickling	X	No indications*	X	-

W indicates the container half with the weld in the cylindrical portion.

* Dye penetrant indications excluding those found visually.

TABLE II (cont.)

Material	Tank Ident.	Exposure Period Months	5-10X Inspection	40X Inspection	40X Weld Inspection	Chemical Analysis	Dye Penetrant Inspection	Metallographic Documentation Macro	Micro
17-4PH SS	P-11W	6	Rough surface where heat treat scale removed by pickling	Metallic granular appearing contamination	Rough surface where heat treat scale removed by pickling	X	No indications*	X	-
17-4PH SS	P-1	6	Rough surface where heat treat scale removed by pickling - rust colored spot in weld	Metallic granular appearing contamination	Rough surface where heat treat scale removed by pickling - crack in weld - rust colored spot in weld	-	No indications*	X	-
17-4PH SS	P-1W	6	Rough surface where heat treat scale removed by pickling	Metallic granular appearing contamination	Rough surface where heat treat scale removed by pickling	-	No indications*	-	-
17-4PH SS	P-12	12	Rough surface where heat treat scale removed by pickling	Metallic granular appearing contamination	Rough surface where heat treat scale removed by pickling	-	No indications*	X	X
17-4PH SS	P-12W	12	Rough surface where heat treat scale removed by pickling	Metallic granular appearing contamination	Rough surface where heat treat scale removed by pickling	-	No indications*	-	-
17-4PH SS	P-2	12	Rough surface where heat treat scale removed by pickling	Metallic granular appearing contamination	Rough surface where heat treat scale removed by pickling	-	No indications*	-	-
17-4PH SS	P-2W	12	Rough surface where heat treat scale removed by pickling	Metallic granular appearing contamination	Rough surface where heat treat scale removed by pickling	-	No indications*	-	-
Inconel 718	1	6	Dark areas at the end of weldments	-	Porous appearance at edge of weld	-	No indications*	X	X

TABLE II (cont.)

Material	Tank Ident.	Exposure Period Months	5-10X Inspection	40X Inspection	40X Weld Inspection	Chemical Analysis	Dye Penetrant Inspection	Metallographic Documentation Macro	Metallographic Documentation M[illegible]
Inconel 718	1W	6	Dark areas at the end of weldments	-	Porous appearance at edge of weld	-	No indications*	-	-
Inconel 718	2	6	Dark areas at the end of weldments	-	Porous appearance at edge of weld	-	No indications*	-	-
Inconel 718	2W	6	Dark areas at the end of weldments	-	Porous appearance at edge of weld	-	No indications*	-	-
Inconel 718	6	12	-	-	Crack in weld	-	No indications*	-	-
Inconel 718	6W	12	-	-	-	-	No indications*	-	-
Inconel 718	4	12	-	-	Crack in weld	-	No indications*	X	X
Inconel 718	4W	12	-	-	-	-	No indications*	-	-
Titanium 6Al-4V	2	6	Spots of titanium oxide from heat treatment (age at 1000°F), baked or stained	Pits and circular knifeline attack in stained areas, titanium oxide	-	-	No indications*	X	X
Titanium 6Al-4V	2W	6	Spots of titanium oxide from heat treatment (age at 1000°F), baked or stained	Pits and circular knifeline attack in stained areas, titanium oxide	-	-	No indications*	-	-
Titanium 6Al-4V	1	6	Spots of titanium oxide from heat treatment (age at 1000°F), baked or stained	Pits and circular knifeline attack in stained areas, titanium oxide	-	-	No indications*	-	-
Titanium 6Al-4V	1W	6	Spots of titanium oxide from heat treatment (age at 1000°F), baked or stained	Pits and circular knifeline attack in stained areas, titanium oxide	-	-	No indications*	-	-
Titanium 6Al-4V	3	12	Spots of titanium oxide from heat treatment (age at 1000°F), baked or stained	Pits and circular knifeline attack in stained areas, titanium oxide	-	-	-	-	-

TABLE II (cont.)

Material	Tank Ident.	Exposure Period Months	5-10X Inspection	40X Inspection	40X Weld Inspection	Chemical Analysis	Dye Penetrant Inspection	Metallographic Documentation Macro	Metallographic Documentation Micro
Titanium 6Al-4V	3W	12	Spots of titanium oxide from heat treatment (age at 1000°F), baked or stained	Pits and circular knifeline attack in stained areas, titanium oxide	-	-	-	-	-
Titanium 6Al-4V	5	12	Spots of titanium oxide from heat treatment (age at 1000°F), baked or stained	Pits and circular knifeline attack in stained areas, titanium oxide	-	-	-	-	-
Titanium 6Al-4V	5W	12	Spots of titanium oxide from heat treatment (age at 1000°F), baked or stained	Pits and circular knifeline attack in stained areas, titanium oxide	-	-	-	-	-

in Figures 16 through 25, with each photomicrograph being shown with its corresponding photomacrograph. The results of the analyses are discussed below for each container material.

a. 304L Stainless Steel

The one discrepancy found in the 304 containers was a small piece of opaque refractory-appearing material in the weld of Containers No. 3 and 5, Figure 16. The small size prevented identification by chemical or instrumental analyses. A metallographic specimen was prepared from No. 3 container; however, most of the material was lost during specimen preparation due to brittleness. In addition, a light red stain appeared adjacent to the closure weld of container No. 11. The cause of the stain could not be determined; its size was 0.15 x 0.01 inch.

b. A-286

A total of four cracks were found in the end closure welds; one crack appearing in Containers No. 13 and A-3, and two cracks appearing in Container No. 6. Three of the cracks are shown in Figures 17 through 19. The cracks appear in both the oxygen-free and oxygen-saturated water containers and are attributed to shrinkage stresses occurring during the welding process. These defects were expected, due to the poor weldability of the A-286 material. The photomicrographs indicate a corrosive attack of either part or all of the weld crack. This behavior is attributed to crevice corrosion during post-heat treatment pickling of the containers. The presence of scale within the crack prior to heat treatment verifies the conclusion that the attack is not a result of water storage. No evidence of corrosion resulting from water storage could be found.

c. 17-4PH (H1025) Stainless Steel

All the containers were in a similar general condition. Examination of the interior surfaces at moderate magnifications revealed that the dark areas shown in Figures 10 and 11 were relief areas on a surface roughened by scale removal during pickling as shown in Figure 20. This examination also revealed that the shiny deposits seen with the unaided eye had the appearance of being metallic and granular as shown in Figure 21. Samples of this material, which adhered tightly to the container inner wall, were examined by emission spectrographic and X-ray diffraction analyses. The sample was identified as the container material, 17-4PH stainless steel. A single weld crack, shown in Figure 22, was found in an end closure weld in Container No. P-1. This crack also occurred during welding and is not a result of the water storage.

Magnification 7X

Magnification 100X

Figure 16. Opaque Appearing Contaminant in Closure Weld of 304 No. 3 Container (Arrows), Section of Weld Taken Through Contaminant (lower Photo). Most of Contaminant was Lost During Sectioning Due to Brittleness.

Magnification 11X

Magnification 200X

Figure 17. Weld Crack in A-286 No. 13 Container. Photomicrograph (right) Shows Heat Treatment Scale in Lower Portion of Crack. Crack at Surface Been Enlarged by Crevice Corrosion During Pickling.

Magnification 20X

Magnification 200X

Figure 18. Weld Crack in A-286 No. 6 Container. Photomicrograph (Bottom) Shows Enlargement of Crack by Crevice Corrosion During Pickling.

Magnification 20X

Magnification 80X

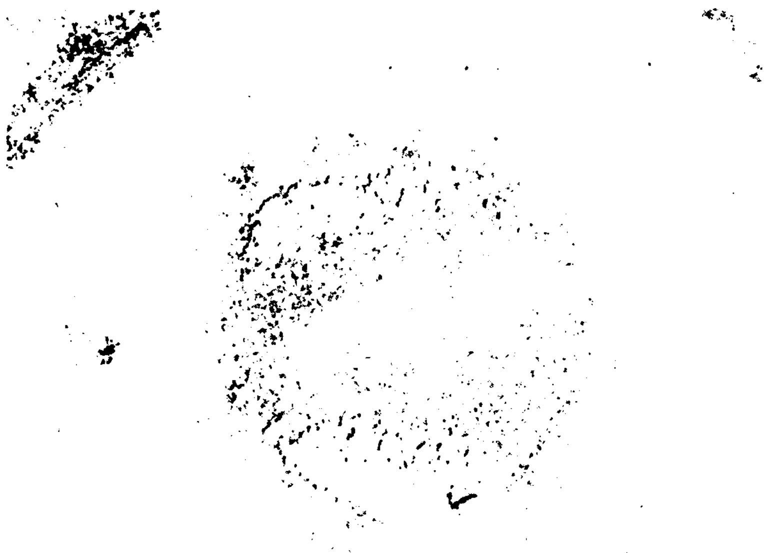
Figure 19. Weld Crack in A-286 No. 3 Container. Photomicrograph (Bottom) Shows Upper Portion of Crack Enlarged Due to Crevice Corrosion During Pickling.

NO. P-1

NO. P-12

Magnification 4X

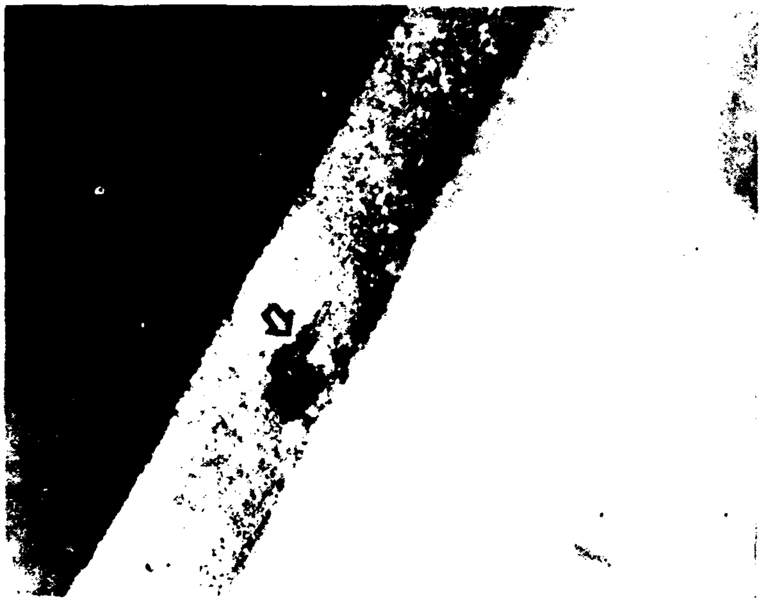
Figure 20. Dark Appearing Areas of Figure 11 are Relief Areas in a Roughened Surface Produced by Scale Removal During Pickling of 17-4 PH Stainless Steel Containers



NO. P-1

NO. P-12

Figure 21. Adherent Metallic Granular Material Found in 17-4PH Stainless Steel Containers. Lower Photo is Bright Area Shown in Figure 11.



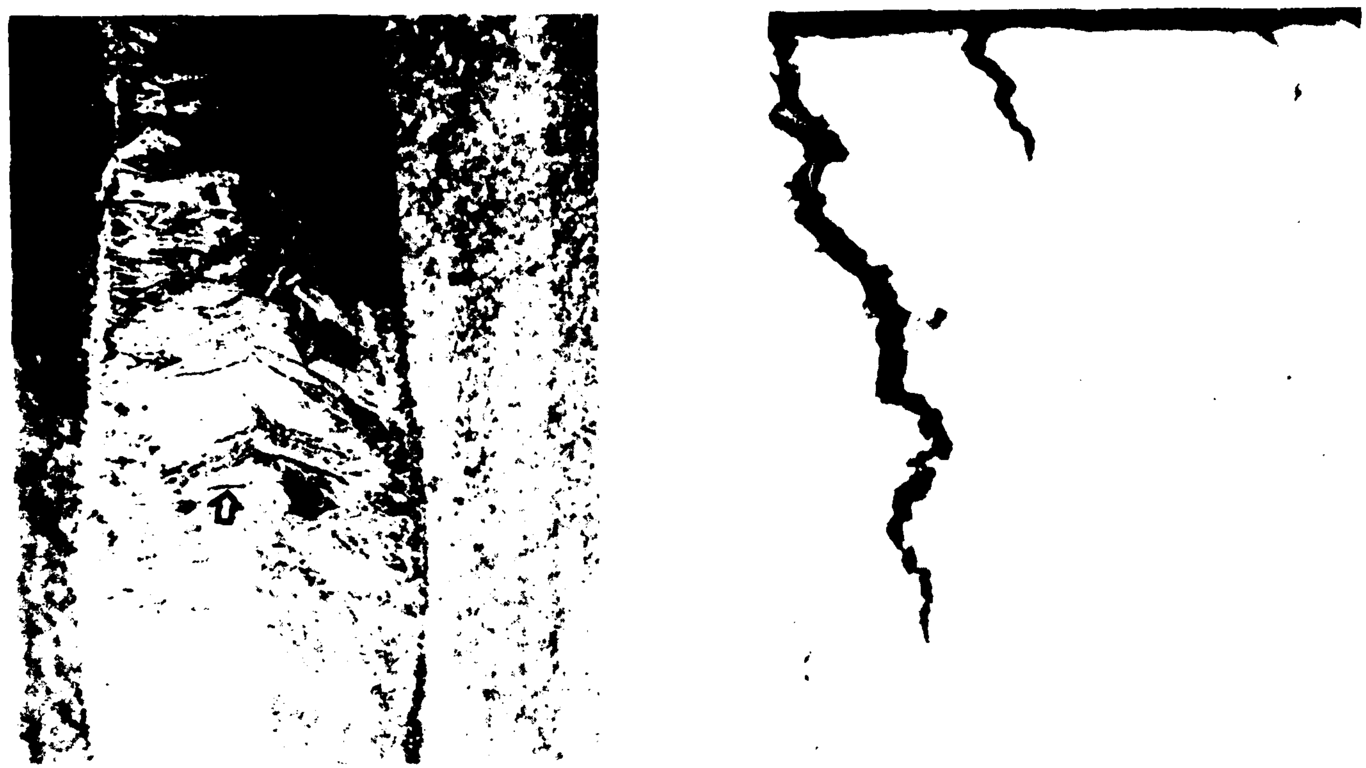
Magnification 10X

Figure 22. Weld Crack in 17-4 PH Stainless Steel No. P-1.

Magnification 20X

Magnification 200X

Figure 23. Dark Porous Appearing Areas at Edge of Welds in Inconel 718 Containers. Photomicrograph (Bottom) Shows Porous Condition.

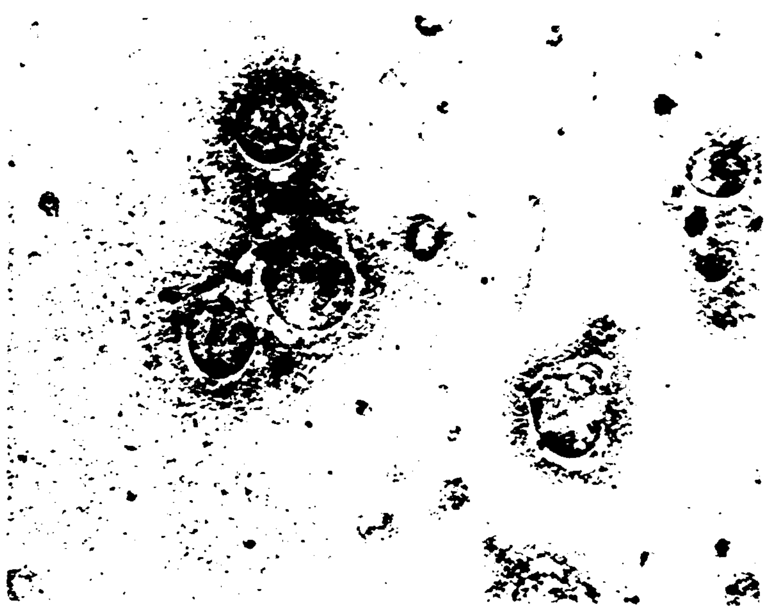


Magnification 21X

Magnification 80X

Figure 24. Weld Shrinkage Cracks in Inconel 718 No. 4 Container (arrows)

Magnification 4X



Magnification 11X

Figure 25. Staining and Corrosion of 6Al-4V Titanium Alloy Containers Resulting From Inadequate Cleaning Prior to Heat Treatment. Top Photo Shows Fingerprint Pattern. Bottom Photo Shows Slight Knife Line Attack at Edge of Stains.

d. Inconel 718

Examination of the interior surfaces at moderate magnification revealed a dark, porous area at the edge of all closure weldments as shown in Figure 23. A photomicrograph showing a section taken through the weld edge is also shown in Figure 23, and a porous condition in the weld immediately adjacent to the parent metal. It could not be definitely established whether the condition is selective attack during pickling, alone, or in combination with entrapment of oxides due to inadequate back-up gas coverage during welding. The condition is not attributable to water storage. Two weld shrinkage cracks were found in the closure welds of Container No. 4 as shown in Figure 24. Again, these defects occurred during container fabrication and did not result from exposure to the stored water.

e. 6Al-4V Titanium

All containers exhibited small isolated areas of titanium oxide from the heat treatment (aging) that were not removed during pickling. Examination of the mottled areas at small magnifications revealed that these areas are primarily stain. Portions of these stained surfaces contained crater-like areas outlined by knifelike corrosion. These discrepancies are shown in Figure 25. Metallographic examination of sections taken through these areas revealed no detectable depth to the corrosion effects associated with the stained areas. Areas where stains appeared in the form of fingerprints were given closer scrutiny to detect possible hot-salt stress corrosion effects, since the containers were aged in the contaminated condition. No defects attributable to water storage were found.

3. Implications of the Results of the Examination

Discrepancies found in the internal surfaces of the containers were due to fabrication and cleaning procedures, and not due to the stored water. The most serious of these discrepancies, with regard to influencing the function of the containers for storing transpiration coolant water, is the generation of a smut on the Inconel 718 and 17-4PH stainless steel and the deposition of parent metal granules on 17-4PH stainless steel during pickling. However, neither of these irregularities have produced particles of sufficient size and quantity to functionally degrade the water quality. The primary cause of these conditions is attributed to poor inert gas coverage on the container interiors during heat treatment and the resulting excessive oxidation. The presence of small amounts of oxide on the titanium alloy cannot be readily explained, because the containers were aged at 1000°F in air and the interiors were pickled for longer times than the exteriors from which all oxides were removed.

The presence of contaminants in the titanium alloy containers prior to heat treatment and the weld cracks found in the A-286, Inconel 718, and 17-4PH stainless steel containers are attributable to inadequate process control on the part of the container supplier. In spite of the presence of the contaminants, the water quality was not significantly impaired.

All discrepancies found on the internal surfaces of the containers were due to fabrication and cleaning procedures and not due to the presence of water in the containers.

SECTION III

CONCLUSIONS AND RECOMMENDATIONS

A. CONCLUSIONS

The following conclusions may be drawn from the twelve-month storage evaluation program.

1. All five container materials, 304 and 17-4PH stainless steels, A-286, Inconel 718, and 6Al-4V titanium, are suitable for the storage of water for transpiration cooling purposes.

2. Both the oxygen-free and oxygen-saturated waters demonstrated favorable storability characteristics with regard to pH changes, ionic contamination as evidenced by electrical resistance measurements, and particulate formation as evidenced by Silting Index values and microscopic inspection of filters.

3. Based on the biological tests, there is no evidence that biological growth has occurred in the containers during storage periods up to twelve months.

4. Discrepancies in the containers such as cracks, smut and etched surfaces were produced during fabrication, cleaning, and passivation of the containers. Yet with adequate flushing prior to storage, the stored water is suitable for use as a transpiration coolant.

B. RECOMMENDATIONS

1. Long-term storage tests should be conducted with the selected waters in containers in which dissimilar metals are present to demonstrate which combinations of metallic materials can be used as suitable components in a transpiration coolant system.

2. The ease of fabrication, cleaning, and passivation of containers should be given proper priority in the selection and design of container materials for transpiration coolant devices.

FABRICATION AND TREATMENT PROCEDURES FOR WATER CONTAINERS

The fabrication procedures, the heat treatment cycles, the cleaning procedures, and the passivation procedures for the water containers prior to filling are presented below.

A. CONTAINER FABRICATION

Drawings of the long term storage container and its associated weld tooling are shown in Figures A-1, A-2, and A-3. The sequence of operations in the container fabrication is as follows:

1. Cylindrical Tube, -1

a. Fabricate -1 cylinder tube by rolling the sheared sheet stock into the desired diameter.

b. Weld the longitudinal joint using the automatic GTA* welding process. Full weld penetration must be obtained using gas backup for the welds.

c. Dye penetrant inspect welds then trim tubes to required length. No cracks allowed.

d. Machine tube ends for weld joint preparation.

e. Clean tubes and store in appropriate container while awaiting for next assembly.

2. Fill Tube, -2

a. Section fill tubes to 6 $\pm$ 0.12 in. lengths.

b. Deburr fill tube ends and inspect.

c. Clean fill tubes and then package individually and store for next assembly.

3. Tank Head, -3

a. Blank tank heads to the desired diameter by punching or sawing and then machining.

b. Machine weld joint preparation at outer edge as required.

c. Drill the fill tube hole to mate with the fill tube.

*Gas Tungsten Arc

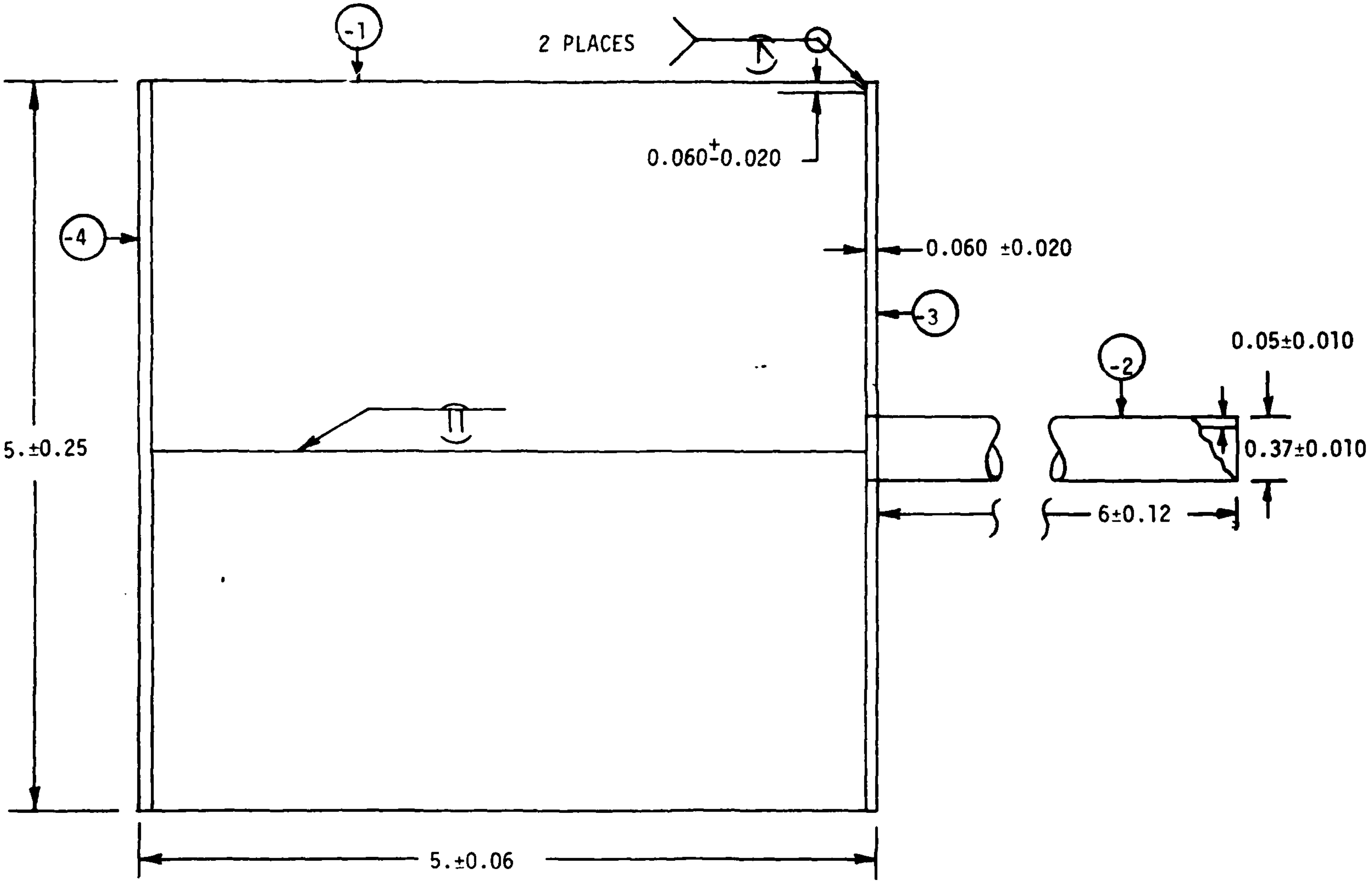


Figure A-1. Long Term Storage Container

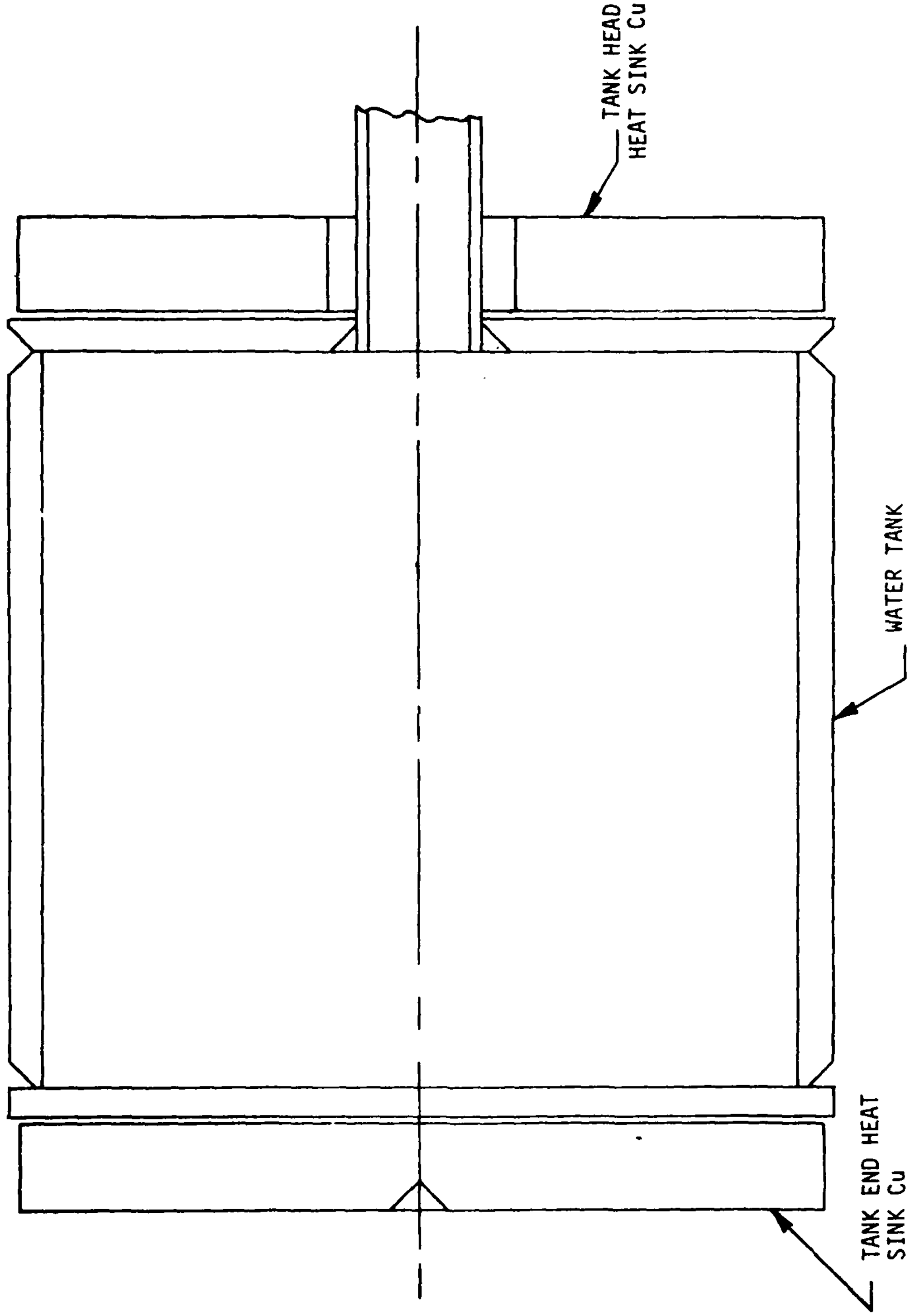


Figure A-2. Weld Tooling for Tank Closures

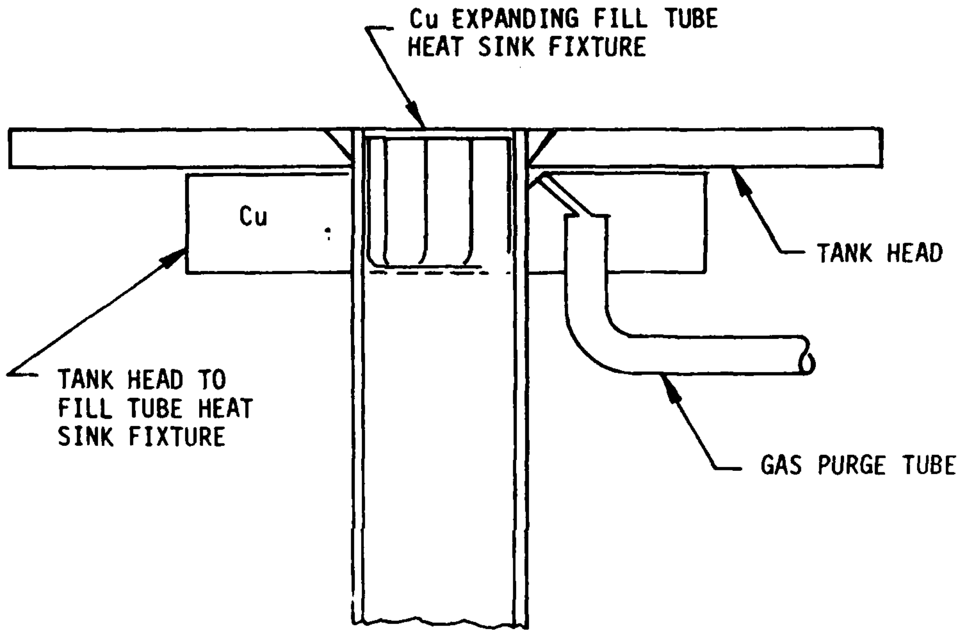


Figure A-3. Weld Tooling for Fill Tube to Tank Head Joint

A, Container Fabrication (cont.)

d. Counter bore the tank head on one side at the fill tube hole for weld joint preparation.

e. Inspect tank heads, clean and package.

4. Tank End, -4

a. Perform operations 3,a, 3,b, and 3,e as above.

5. Assembly Sequence

a. Assemble -2 fill tube into -3 tank head using appropriate weld fixture and tooling.

b. Weld root pass on grooves side without weld filler wire by the automatic GTA welding process.

c. Clean root pass by rotary wire brushing using a clean stainless steel wire brush.

d. Weld the cover pass using the appropriate weld filler wire.

e. Reposition the part in the weld fixture and then place a fillet weld at the fill tube to tank head junction.

f. Inspect welds and clean part.

g. Fixture tank head assembly with -1 tube for welding and purge tank with Ar or He.

h. Weld root pass without the use of weld filler wire. Full penetration must be obtained at the ID of the joint.

i. Dye penetrant inspect and clean root pass. No cracks allowed.

j. Fill weld groove using the appropriate weld filler wire.

k. Inspect and clean the inner tank surfaces.

l. Locate the tank end to the tank for welding, then purge the closed tank with Ar or He.

m. Make the root pass without the use of weld filler wire to insure full penetration.

n. Clean root pass surface.

o. Inspect the internal weld penetration using a borescope. Full weld penetration is required.

p. Run weld cover pass using the appropriate weld filler wire.

q. Clean, inspect, and package unit for shipment.

Heat treatment of the containers was performed in an argon atmosphere and with one exception, as noted below, utilizing the thermal cycles listed in Table A-I.

Upon receipt of the tanks, the certifications were examined and it was found that some of the material used to fabricate the 304L stainless steel containers was actually 304 stainless steel. With the concurrence of the Air Force, the containers were subjected to a heat-treatment at 1925°F in hydrogen, followed by rapid cooling to preclude sensitization in the weld heat affected zone.

B. PREPARATIVE PROCEDURES

1. Container Cleaning and Passivation

All the containers were degreased by submerging and agitating the tanks three times in fresh isopropyl alcohol. The containers were then purged with dry, filtered nitrogen and placed in a vacuum chamber for final drying. The A-286, 17-4PH stainless steel, and Inconel 718 containers were then subjected to an alkaline descaling treatment for 60 minutes with Kelite No. 235 at a concentration level of 32 oz per gal at 190°F. The containers were then rinsed with water at 150°F for 2 to 5 minutes, followed by a rinse at ambient temperatures with deionized water for 2 to 5 minutes, then purged with dry, filtered nitrogen, and finally dried in a vacuum chamber.

The 304, A-286, and 17-4PH stainless steels and Inconel 718 were descaled in a pickling solution of 20% HNO_3, 5% HF, and 75% H_2O at 130°F for 30 minutes per immersion until the last traces of scale were removed as confirmed by examination with a borescope. Following the acid treatment, the tanks were flushed with tap water for 2 to 5 minutes, then flushed with deionized water for 2 to 5 minutes, then subjected to ultrasonic vibration in a water bath for 15 minutes, flushed with deionized water for 5 minutes, purged with dry, filtered nitrogen, and finally dried in a vacuum chamber.

TABLE A-I

HEAT TREATMENT PROCEDURES FOR WATER CONTAINERS

Material	Weld Wire Material	Heat Treatment	Surface Finish
304L Stainless Steel	-	-	Alkaline Clean, Pickle and Passivate
304L Stainless Steel	308L	-	Alkaline Clean, Pickle and Passivate
A-286	-	Solution Treat and Age - MIL-H-6875	Alkaline Clean, Pickle and Passivate
A-286	A-286	Solution Treat and Age - MIL-H-6875	Alkaline Clean, Pickle and Passivate
17-4 PH Stainless Steel	-	Age to H-1025 Condition - MIL-H-6875	Alkaline Clean, Pickle and Passivate
17-4 PH Stainless Steel	17-4	Solution Treat and Age to H-1025 Condition - MIL-H-6875	Alkaline Clean, Pickle and Passivate
Inconel 718	-	Age (1400 - 1200°F)	Alkaline Clean, Pickle and Passivate
Inconel 718	Inconel 718	Solution Treat (1950°F) and Age (1400-1200°F)	Alkaline Clean, Pickle and Passivate
6Al-4V-Titanium	6Al-4V Titanium	Age (1000°F - 4 Hours) - Weld-Stress Relieve (1000°F - 4 Hours)	Pickle

B, Preparative Procedures (cont.)

The 304 and A-286 stainless steels, and Inconel 718 were passivated with a 30% HNO_3/3% $Na_2Cr_2O_7$ aqueous solution at 130°F for 25 minutes, rinsed with tap water for 2 to 5 minutes, rinsed with deionized water for 2 to 5 minutes, subjected to ultrasonic vibration in water for 5 minutes, rinsed with deionized water for 2 to 5 minutes, purged with dry, filtered nitrogen, and dried in a vacuum chamber. The 17-4PH stainless steel received the same treatment with the exception of a 10 minute passivation time.

The 6Al-4V titanium was degreased in isopropyl alcohol as described previously for the other tankage and then descaled in a pickling solution of 33.2% HNO_3, 1.6% HF, and 65.2% water at 140°F for 3 minutes. The titanium containers were rinsed with 130°F tap water for 2 to 5 minutes, followed by a rinse in deionized water for 2 to 5 minutes, then subjected to ultrasonic vibration in water for 5 minutes, rinsed with deionized water for 2 to 5 minutes, purged with dry nitrogen, and then dried in a vacuum chamber.

2. Container Sterilization, Filling, and Sealing

Following the final rinsing with deionized water and the drying of the container in a vacuum chamber, the containers were wrapped with reusable sterilization paper. The wrapped containers were then sterilized in an autoclave at 250°F with 15 psig steam for 30 minutes followed by a 30-minute drying period. The containers were then stored in the paper to maintain their sterile condition.

All the steps required to fill the containers with water were conducted in the sterile laminar flow bench. The tanks were removed from the wrapping paper in the laminar flow bench. The tanks were weighed empty; then weighed when filled completely with sterile water to determine the total volume of the tank. The water was drained out and the tank was rinsed once more with the sterile water. A sample of the rinse water was checked for pH, conductivity, and Silting Index. If the values indicated that particulate matter and dissolved species were not present, the tank was considered ready for filling; if the values indicated that contaminants were present, the tank was rinsed until there was no evidence of contamination.

Before the final filling with oxygen-saturated deionized water, the tank was purged with oxygen from a filtered supply. The tank was then filled with the water and a sample was withdrawn for pH, conductivity, and Silting Index measurements. The ullage was adjusted to the ten percent value by weighing the container and its contents; the ullage space was purged with the filtered oxygen; and the container was capped with a sterile, tapered plug made from the same material as the container. The plug was seated in the fill-tube by use of a hammer. The containers were filled with the oxygen-free, deionized water in an analogous manner except that filtered nitrogen was used instead of oxygen for purging and blanketing the container.

B, Preparative Procedures (cont.)

The final sealing of the containers was accomplished by GTA welding the fill-tube/plug interface. The welds were inspected visually for any apparent anomalies. None were found. Then the containers were placed in plastic bags and labeled for the long-term storage tests.

The sampling plan for the long-term storage tests is to remove one container of each material with the two types of water for inspection and evaluation every six months for a period of five years. The contents will be characterized with respect to pH, conductivity, particulate content, and biological activity; and the containers themselves will be subjected to metallurgical examination if the other test data indicate that this is required.

SILTING INDEX MEASUREMENT

In order to obtain a measurement of the clogging tendencies of the particulate matter which may be produced during the storage of water in the presence of metals and non-metals for prolonged periods of time, a flow test of the water samples through nominal 1 micron size pores was required. Due to the limited quantity of the water available for each sample, approximately 20 ml, the ASTM Method F 52-69, "Silting Index of Fluids for Processing Electron and Micro-electronic Devices", was selected as being appropriate for the program.

The method determines the silting or clogging tendency of a fluid containing fine particles and gelatinous materials suspended in the fluid. The fluid is filtered through a membrane filter having a uniform pore size of 0.8 micron at a constant differential pressure. Particles larger than 5 microns form an open network above the filter and do not affect the clogging tendency; particles smaller than 5 microns tend to block the flow passages of the filter and cause a decay in the flow rate. The rate of flow decay is expressed in terms of a Silting Index value; the greater the value, the greater the clogging tendency. A schematic diagram of the apparatus is shown in Figure B-1.

The total volume of fluid passed through the filter is 12 ml. The flow of the last 10 ml is timed incrementally as V_1 (1 ml), V_2 (5 ml), and V_3 (10 ml) with T_1, T_2, and T_3 the times required to flow the respective volumes. The Silting Index value is calculated from the equation:

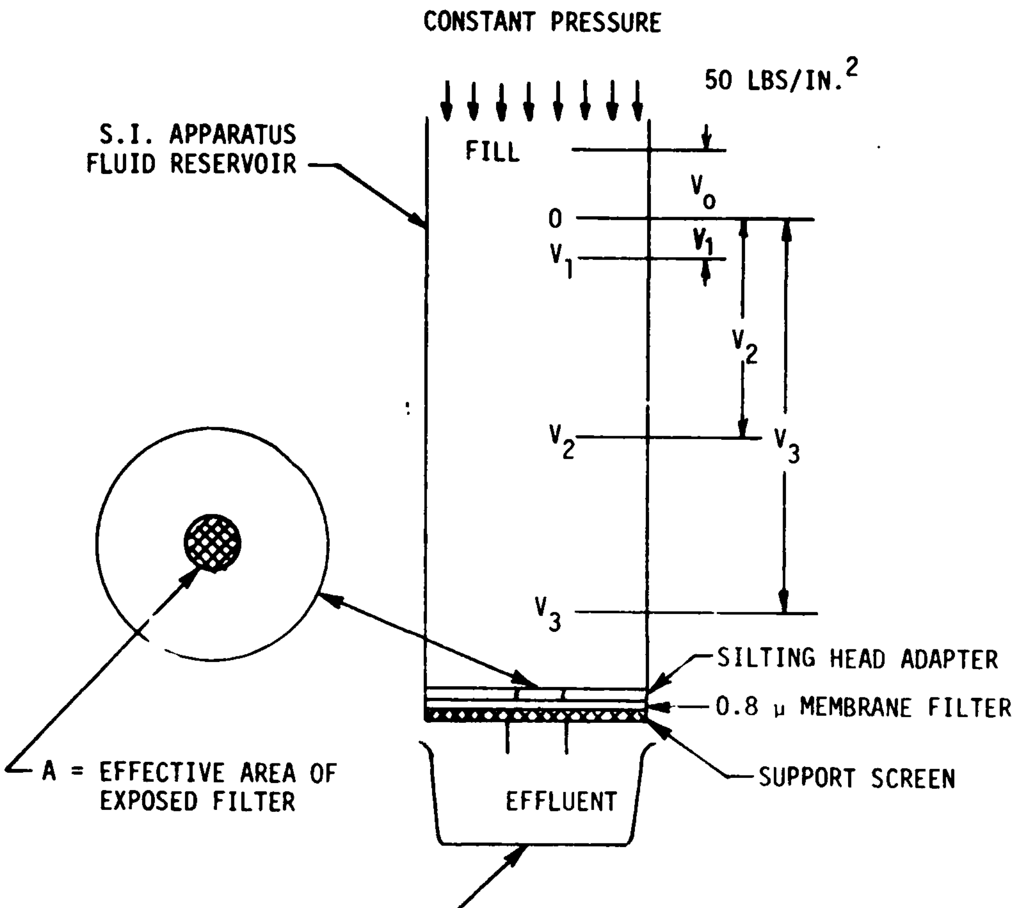
$$S.I. = \frac{T_3 - 2T_2}{T_1}$$

The tests in the program were conducted with three silting heads, No. 1 with an effective area of 1.0 mm^2, No. 2 with an effective filter area of 4.3 mm^2, and No. 3 with an effective filter area of 18.5 mm^2. The procedure used in the tests was that prescribed in ASTM Method F52-69 except that samples were tested in triplicate only when sufficient sample was available. A photograph of the apparatus* used is shown in Figure B-2.

Water which had passed through the 0.22 micron pore size absolute filter and which was used in preparation of the tests, always produced a Silting Index value of less than 1 using the No. 1 silting head which has a cross-sectional area of 1 mm^2. The effect of particle size on the Silting Index was evaluated using latex spheres with a mean particle size of 1.25 microns with a range from 0.5 to 2.0 microns and puff ball spores with a size range from 3 to 4 microns. These data are presented for silting heads Nos. 1, 2, and 3 which have cross-sectional areas of 1 mm^2, 4.3 mm^2, and 18.5 mm^2, respectively, in Figures B-3 and B-4. The particle concentration is given in mg/1 because the suspensions were prepared on a weight basis; the actual

---

*Available from Millipore Corp., Bedford, MA.



VOLUME - RATIO

$V_o/V_1 = 2/1$

$V_2/V_1 = 5/1$

$V_3/V_1 = 10/1$

Figure B-1. Schematic of Silting Index Apparatus

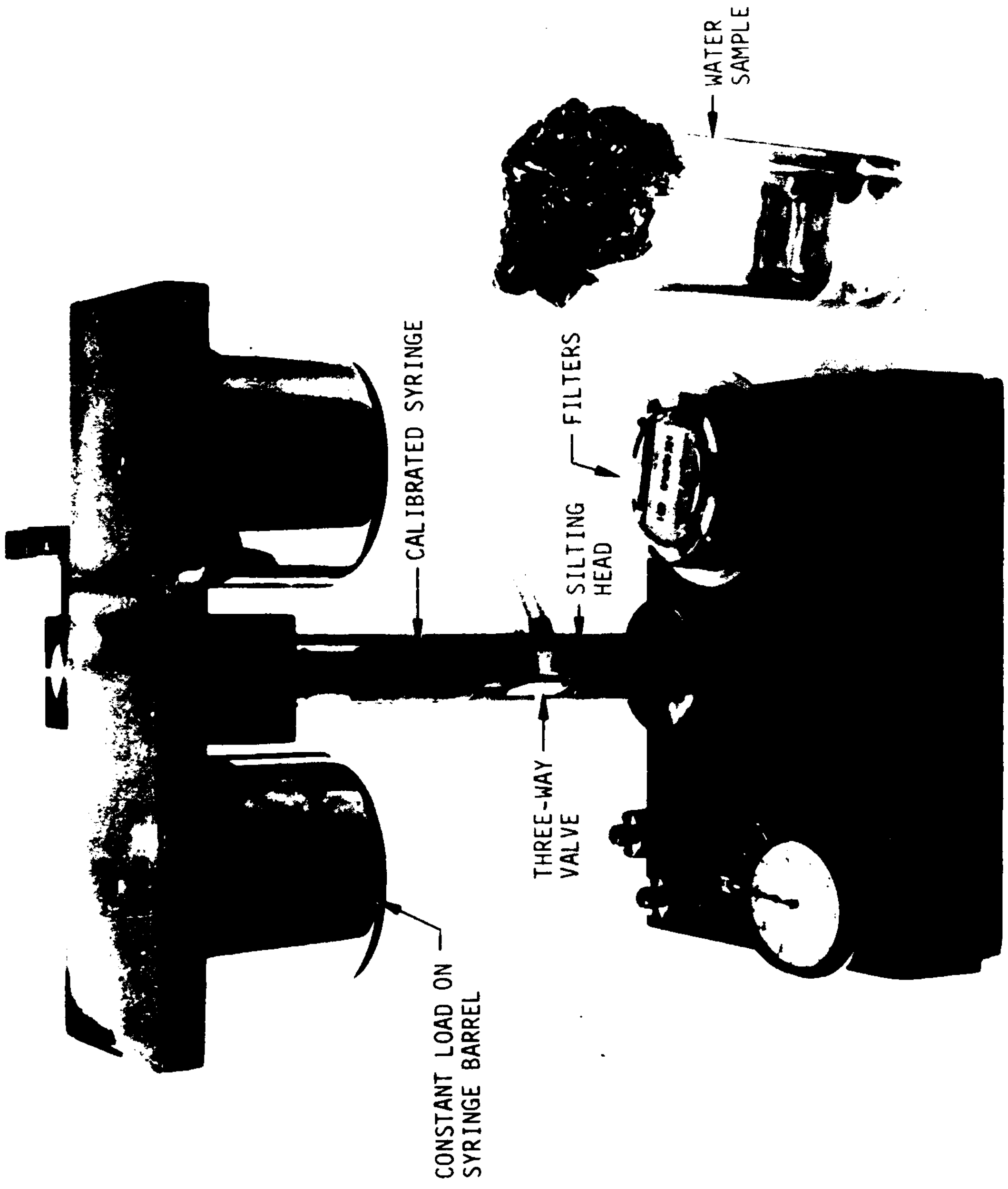


Figure B-2. Silting Index Apparatus

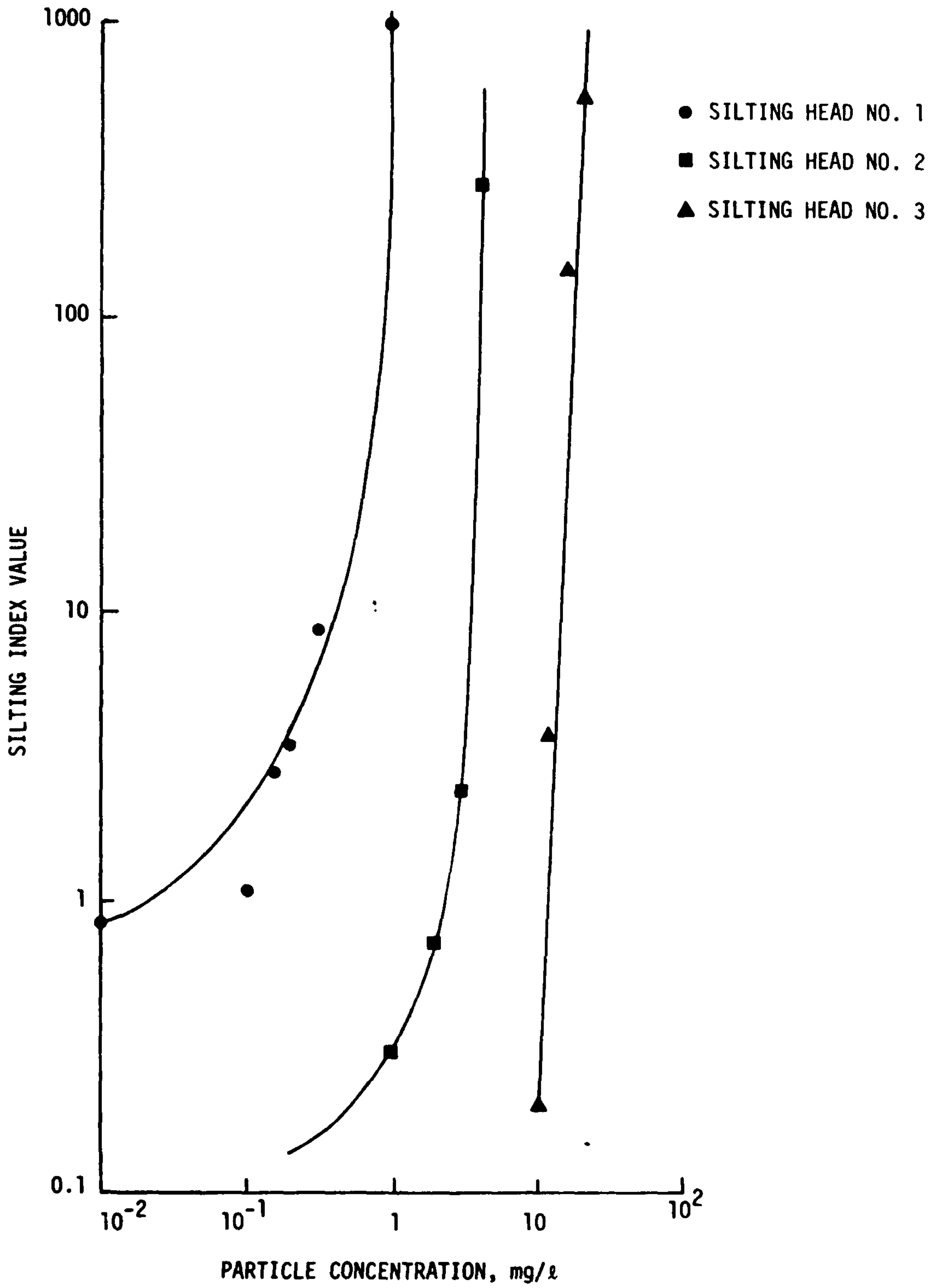


Figure B-3. Concentration Effect of 1.25 Micron Average Diameter Latex Particles on the Silting Index Values

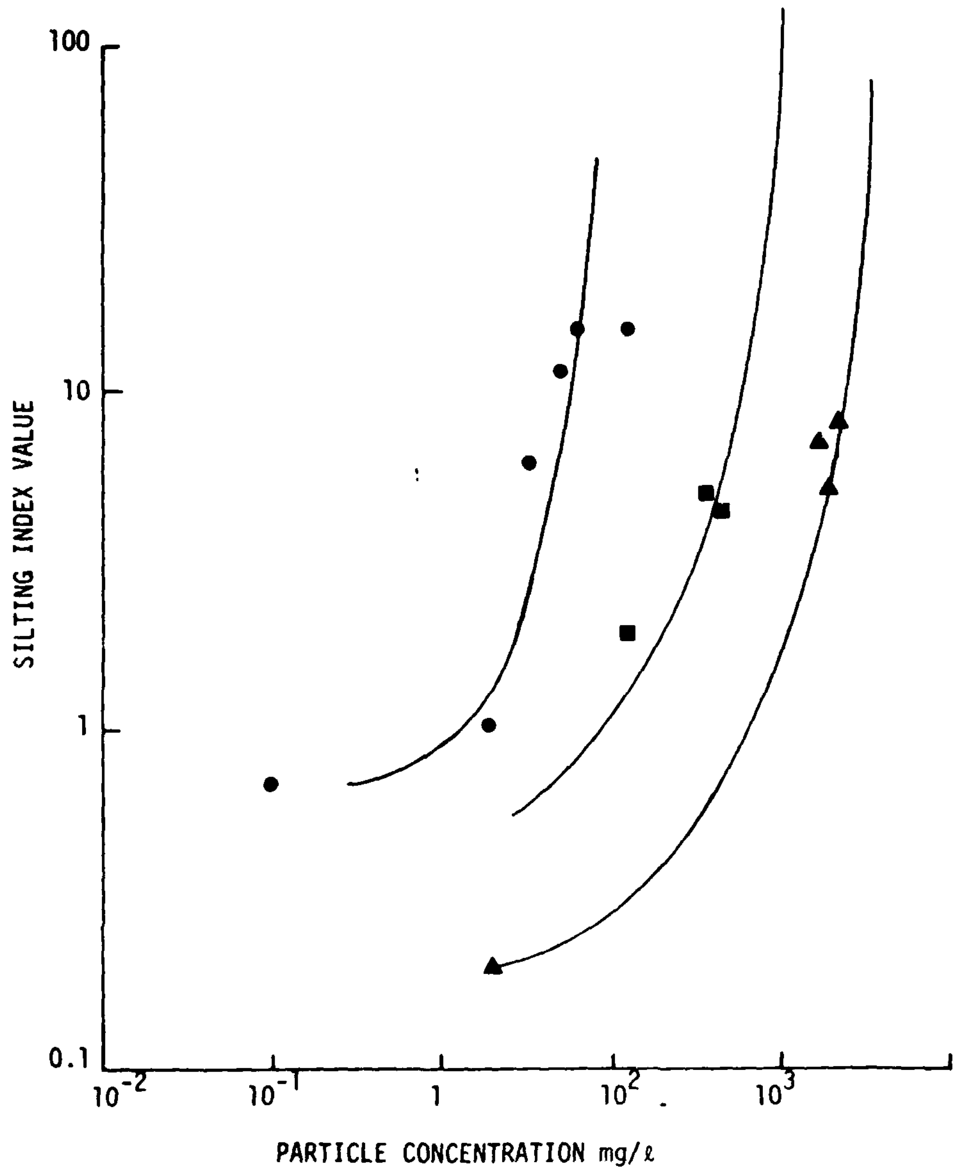


Figure B-4. Concentration Effect of 3-4 Micron Diameter Puff Ball Spores on the Silting Index Value densities of the particles are comparable. The significant item to note from the comparison of the data is that the Silting Index values are comparable at concentration levels that differ by at least one order of magnitude, the smaller particle producing the higher Silting Index value.